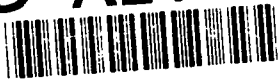


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STATE UNIVERSITY SYSTEM OF FLORIDA



Florida Initiative

ADVANCED MICROELECTRONICS AND MATERIALS PROGRAM

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Advanced Microelectronics and Materials Program

FINAL REPORT

1 July 1988-30 September 1991

DARPA Grant No. MDA972-88-J-1006

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1 July 1988 - 30 September 1991

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19. Abstract

The Advanced Microelectronics and Materials Program (AMMP) has successfully demonstrated the effectiveness of the distributed center cooperative research model for multi-university research by producing outstanding technical accomplishments. Seven state universities within the State University System (SUS) of Florida, namely

Florida A&M University (FAMU)
Florida Atlantic University (FAU)
Florida International University (FIU)
Florida State University (FSU)

University of Central Florida (UCF)
University of Florida (UF)
University of South Florida (USF)

and the sixteen subcontractors located throughout the U.S. have collaborated successfully to perform this program.

The AMMP program has been jointly funded by the Defense Advanced Research Projects Agency (DARPA) and the State University System (SUS) of Florida. The program was initiated 1 July 1988 and successfully completed all objectives on 30 September 1991.

The primary AMMP objective was to plan and to implement productive applied research in four critical technology areas: Microelectronics, Optoelectronics, Superconductivity, and Composite Materials. The results of the research program have far exceeded expectations. The research activity which is briefly summarized in this final report has produced numerous significant accomplishments as documented by over 600 technical publications and twenty-seven patent applications.

A corollary objective of effective technology transfer to industry has also been very successfully accomplished. The team emphasis on transferring the wide-ranging technical results to industry has now produced over eighty industry collaborations. The nation's competitiveness has been enhanced and new devices, products, processes, and systems are under development in industry as a direct result of AMMP and the technology transfer program.

The AMMP program has made a substantial difference in further improving the research capability of the State University System of Florida. As a result of AMMP and the SUS matching funds, the SUS has added over 30 new research faculty. Additionally, a number of new research labs and facilities have been built and over \$15 million in state-of-the-art research equipment has been added.

Research Accomplishments

The extensive new equipment and facilities which have been installed during the first and second year are now fully operational. Their availability has greatly enhanced the progress on the research agenda in the four areas. Specific details are briefly summarized in the following paragraphs.

Microelectronics research has produced significant advances in Ultra Large Scale Integration (ULSI) or Wafer Scale Integration (WSI), high-speed test, and in the new defect engineering project. In ULSI and WSI, new approaches in architecture, test methodology, and interconnect technologies were developed through the Alpha-Site project and the programmable systolic data processor project and the results transferred to industry. The high speed test project has not only demonstrated gigahertz functional testing capability but also 100 MHz single die functional testing. In the defect engineering area, a potentially significant new method for non-destructive, non-contact measurement of iron concentration in silicon with sensitivity of 1 part per trillion has been developed.

Optoelectronics research has produced new world records in the performance of both semiconductor and solid state lasers, semiconductor detectors, and non-linear optical materials.

The world's first stable p-type doping of zinc selenide (ZnSe) with the world's record low resistivity of $0.75 \Omega\text{-cm}$ was demonstrated. This technology has been transferred to the 3M Co. and has resulted in the first demonstration of a ZnSe electrically injected diode laser operating in the blue region at liquid nitrogen temperature and the green region at room temperature. In addition to the first electrically injected ZnSe laser, the team also demonstrated the world's first optically pumped lasing of ZnSe at room temperature. Indium gallium phosphide epitaxial layers were grown and processed to demonstrate light emitting diodes operating at 580 to 603 nm. This technology was transferred to EPITAXX.

In the area of semiconductor devices, indium gallium arsenide layers were heteroepitaxed to gallium arsenide substrates and photodetectors operating at $1.55\mu\text{m}$ with dark currents less than 10^{-6} A were demonstrated. A photodetector operating at 840nm with a world record responsivity-bandwidth product was designed and demonstrated from aluminum gallium arsenide. Multiple quantum wells and superlattices were grown and used to demonstrate tunability of photodetectors from near $1\mu\text{m}$ to over $10\mu\text{m}$. Dual wavelength photodetectors at wavelengths of about $10\mu\text{m}$ were also demonstrated.

A number of bulk crystals for solid state lasers and non-linear optical response were grown using both high temperature melt and low temperature solution growth techniques. A milestone achievement was the growth of chromium doped lithium strontium aluminum fluoride crystals of sufficient quality to cut and polish optical cavities and demonstrate flash-lamp pumped lasing from 780nm to 1040nm. Output from this crystal was frequency doubled to demonstrate blue emission at high power with good efficiency. Simplified growth procedures for neodymium doped yttrium lithium fluoride crystals were successfully transferred to Lightning Optical.

Achievements in superconductor synthesis and processing covered the full range of methods and materials, and provided some novel new techniques. A non line of sight modification of laser ablation has yielded T_c 's and critical currents at the highest reported values. The early difficulties in obtaining good performance from sputtered films was found to be due to negative oxygen ion resputtering, and a correction for this problem has been identified. Free standing superconducting films, of potential value in bolometer performance, were achieved first, and only, in this program. Modulated structures of YBaCuO/PrBaCuO were produced showing that epitaxial insulating layers of thickness down to 20\AA could be grown over equally thin superconducting 123 layers while retaining T_c 's $> 60\text{K}$ - an important step in the search for a Josephson device barrier. Barrier layers to successfully prevent diffusion into both the superconducting material, and device silicon, have been found. Finally, a new, simple method has been developed to produce patterned films onto transparent substrates in a single step, and which can be performed in an open room (nonvacuum) environment (patent applied for).

The Advanced Composite Materials program has produced significant advances in the processing, properties and characterization of composites for ultra high temperature applications. The overall objective of this program was to provide a fundamental understanding of the processing science and technology necessary to fabricate ceramic-matrix, intermetallic-matrix, and metal-matrix composites with superior mechanical properties in high temperature and oxidizing environments. The composites are intended for use as structural materials for advanced aerospace applications at temperatures exceeding 1200°C (2200°F). The program consisted of four interactive research projects in the areas of Fiber Fabrication, Coatings and Infiltration, Composite Fabrication, and Physical/Mechanical Properties. Significant accomplishments were achieved in each area. The programs have also led to extensive research collaboration with several industries, as well as other universities.

Acknowledgements

The Advanced Microelectronics and Materials Program team would like to sincerely thank the DARPA and ONR project and contract officers:

Mr. Sven Roosild, DARPA Project Officer
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Mr. Scott Ulrey, DARPA Contract Officer

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Introduction and Summary

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University of Central Florida (UCF)
University of Florida (UF)
University of South Florida (USF)

and the following sixteen subcontractors located throughout the U.S. have collaborated successfully to perform this program:

UF subcontractors:	Arizona State University (ASU) The University of Arizona (UOA) The University of Illinois (ILL) Kansas State University (KSU) Mercer University (Merc) Massachusetts Institute of Technology (MIT) The University of California, Santa Barbara (UCSB) TRW Incorporated
USF subcontractors:	Boston University (BU) Carnegie-Mellon University (CMU) Florida Institute of Technology (FIT) The University of Michigan (Mich) The University of Southern California (USC)
UCF subcontractors:	Stanford University (Stan) Massachusetts Institute of Technology (MIT-UCF)
FIU subcontractors:	Coors Incorporated

The AMMP program has been jointly funded by the Defense Advanced Research Projects Agency (DARPA) and the State University System (SUS) of Florida. The program was initiated 1 July 1988 and successfully completed all objectives on 30 September 1991.

The primary AMMP objective was to plan and to implement productive applied research in four critical technology areas: Microelectronics, Optoelectronics, Superconductivity, and Composite Materials. The results of the research program have far exceeded expectations. The research activity which is briefly summarized in this final report has produced numerous significant accomplishments as documented by over 600 technical publications and twenty-seven patent applications.

A corollary objective of effective technology transfer to industry has also been very successfully accomplished. The team emphasis on transferring the wide-ranging technical results to industry has now produced over eighty industry collaborations. The nation's competitiveness has been enhanced

and new devices, products, processes, and systems are under development in industry as a direct result of AMMP and the technology transfer program.

The AMMP program has made a substantial difference in further improving the overall research capability of the State University System of Florida. As a result of AMMP and the SUS matching funds, the SUS has added over 30 new research faculty. Additionally, a number of new research labs and facilities have been built and over \$15 million in state-of-the-art research equipment has been added.

The new research faculty and labs and equipment were significant factors in the competitive award in 1990 of the National High Magnetics Field Laboratory by the National Science Foundation. The winning team headed by Florida State University included the University of Florida and the Los Alamos National Laboratory.

Four state-funded "centers of excellence" contributed significantly to the AMMP program, namely:

CMR	-	The Center for Microelectronics Research University of South Florida, Tampa
CREOL	-	The Center for Research in Electro-Optics and Lasers University of Central Florida, Orlando
MARTECH	-	Florida State University, Tallahassee
MICROFABRITECH	-	University of Florida, Gainesville

These four "centers of excellence" were making rapid progress prior to AMMP, but with the addition of the new research faculty, the extensive new laboratories and equipment and the significant research accomplishments as a result of AMMP, they have now gained national recognition in their respective fields.

The AMMP team has placed a concerted effort on pursuing competitive R & D funding opportunities in the four technology areas during the second and third year of the program. This effort has resulted in the award of over \$10 million in new funding and is permitting the orderly continuation of key projects and research teams at the participating universities. A number of additional proposals have been submitted and are under evaluation at the time of this report.

Reporting Activities

This final report, in the requested executive summary format, covers the entire AMMP program from 1 July 1988 through the current no-cost extension to completion on 30 September 1991. More extensive technical details are contained in the two prior annual reports for the periods ending 30 September 1989 and 31 December 1990 which are listed in the section on references.

Two annual program reviews were also conducted. The first, held 16-18 January 1990 was highly successful and AMMP was commended for overall accomplishments during the initial year. The DARPA review team was particularly pleased with the quality of the research teams, the new equipment and facilities, and the technical achievements. They commented also on the effectiveness of the distributed center research model and on the high degree of multi-university cooperation. Program participants were encouraged to place a major emphasis on industrial collaboration and

technology transfer; and to submit responses to DoD competitive technology initiatives during the second year.

The second annual review held 16-18 October 1990 was considered even more successful than the first. The review featured more in-depth technical presentations on selected research projects. The informal review comments were very positive. The AMMP team was encouraged by the feedback both from the government review team and the large number of prominent industry representatives that attended the three-day review.

Additionally, the AMMP team placed considerable emphasis on less formal methods of reporting on the wide range of activities. Six technical newsletters were published bi-monthly during the second year and received favorable comment from the wide government, university, and industry distribution. The AMMP team also published three brochures containing summary results and extensive photographs of new equipment, facilities, and research results. A brief 35 minute video tape covering research activities at all seven state universities was also produced.

Research Accomplishments

The extensive new equipment and facilities which have been installed during the first and second year are now fully operational. Their availability has greatly enhanced the progress on the research agenda in the four areas. Specific details are briefly summarized in the following paragraphs. More detailed coverage of the extensive accomplishments is included in the summary sections within the body of this report and the extensive bibliography of published papers or preprints contained in the enclosed References and Publications section.

MICROELECTRONICS

Microelectronics research has produced significant advances in Ultra Large Scale Integration (ULSI) or Wafer Scale Integration (WSI), high-speed test, and in the new defect engineering project. In ULSI and WSI, new approaches in architecture, test methodology, and interconnect technologies were developed through the Alpha-Site project and the programmable systolic data processor project and the results transferred to industry. The high speed test project has not only demonstrated gigahertz functional testing capability but also 100 MHz single die functional testing. In the defect engineering area, a potentially significant new method for non-destructive, non-contact measurement of iron concentration in silicon with sensitivity of 1 part per trillion has been developed.

AMMP program accomplishments are attributed not only to the excellent research teams, but also to the first year investment in a unique facility and equipment infrastructure. In particular, the investment in a very fine geometry laser table and also a state-of-the-art cleanroom has produced excellent research in the area of interconnect technologies for both monolithic and hybrid wafer scale integration. Specific research areas include polyamide milling, direct laser write for process masking, and plated-through holes in silicon for z-axis interconnects. Three industrial projects, which take direct advantage of this research, are presently underway.

The investment in a 200 MHz Hewlett Packard HP82000 tester has produced a test facility that is unmatched in other U.S. universities. This test facility has produced significant industrial interaction and opportunity for technology transfer. Hewlett Packard sponsored an annual international HP82000 users group meeting at USF/CMR in 1990 attended by more than seventy people from more than twenty companies and government agencies. The 1991 meeting was also held at

USF/CMR on April 4-5, 1991. The test facility has demonstrated single die testing over 100 MHz and functional test capability of VLSI devices over a gigahertz. Both tests are considered significant accomplishments. Several industrial programs are now centered around these test facilities and their high speed test capabilities.

Major upgrades were also made to the USF/CMR Microelectronics Design Laboratory. The hardware and software tools in the CMR Microelectronics Design Laboratory now include a complete set of state-of-the-art commercial design automation tools from Cadence, as well as tools especially developed for Wafer Scale Integration Design and reconfiguration by Lincoln Laboratory-SLASH (Standard Linking Automation SHell). CMR has also recently acquired signal processing systems modeling tools from COMDISCOTM. This set of tools has been used by CMR in the development of its current wafer scale designs.

A new laboratory in defect engineering has also been established at USF/CMR with extensive equipment for the measurement of defects and their electronic properties, in silicon, SOI, and III-V compounds. Initial research efforts have focused on the study of properties and characteristics of heavy metals in silicon. Initial results on new methods for non-destructive, non-contact measurements in silicon could provide significant cost savings to industry in terms of increased yield.

OPTOELECTRONICS

Optoelectronics research has produced new world records in the performance of both semiconductor and solid state lasers, semiconductor detectors, and non-linear optical materials.

The world's first stable p-type doping of zinc selenide (ZnSe) with the world's record low resistivity of 0.75 Ω -cm was demonstrated. This technology has been transferred to the 3M Co. and has resulted in the first demonstration of a ZnSe electrically injected diode laser operating in the blue region at liquid nitrogen temperature and the green region at room temperature. In addition to the first electrically injected ZnSe laser, the team also demonstrated the world's first optically pumped lasing of ZnSe at room temperature. Indium gallium phosphide epitaxial layers were grown and processed to demonstrate light emitting diodes operating at 580 to 603 nm. This technology was transferred to EPITAXX. These advances were achieved with a vertically integrated compound semiconductor research program focused on advancement of wide bandgap materials. The vertical integration provided rapid characterization of materials and devices to quickly improve materials growth and processing. This allowed dramatic advances in the use and demonstration of new devices from new materials.

In the area of semiconductor devices, indium gallium arsenide layers were heteroepitaxed to gallium arsenide substrates and photodetectors operating at 1.55 μ m with dark currents less than 10^{-6} A were demonstrated. A photodetector operating at 840nm with a world record responsivity-bandwidth product was designed and demonstrated from aluminum gallium arsenide. Multiple quantum wells and superlattices were grown and used to demonstrate tunability of photodetectors from near 1 μ m to over 10 μ m. Dual wavelength photodetectors at wavelengths of about 10 μ m were also demonstrated. Waveguides in semiconductor materials were shown to be suitable for optical switching and optical logic functions.

A number of bulk crystals for solid state lasers and non-linear optical response were grown using both high temperature melt and low temperature solution growth techniques. A milestone achieve-

ment was the growth of chromium doped lithium strontium aluminum fluoride crystals of sufficient quality to cut and polish optical cavities and demonstrate flash-lamp pumped lasing from 780nm to 1040nm. Output from this crystal was frequency doubled to demonstrate blue emission at high power with good efficiency. Simplified growth procedures for neodymium doped yttrium lithium fluoride crystals were successfully transferred to Lightning Optical.

SUPERCONDUCTIVITY

Achievements in superconductor synthesis and processing covered the full range of methods and materials, and provided some novel new techniques. A non line of sight modification of laser ablation has yielded T_c s and critical currents at the highest reported values. The early difficulties in obtaining good performance from sputtered films was found to be due to negative oxygen ion resputtering, and a correction for this problem has been identified. Free standing superconducting films, of potential value in bolometer performance, were achieved first, and only, in this program. Modulated structures of YBaCuO/PrBaCuO were produced showing that epitaxial insulating layers of thickness down to 20Å could be grown over equally thin superconducting 123 layers while retaining T_c s $> 60K$ - an important step in the search for a Josephson device barrier. Barrier layers to successfully prevent diffusion into both the superconducting material, and device silicon, have been found. Finally, a new, simple method has been developed to produce patterned films onto transparent substrates in a single step, and which can be performed in an open room (nonvacuum) environment (patent applied for).

In characterization and properties, we have applied many techniques to determine the nature, quality, and performance of our materials including the development of new methods. Persistent current lifetimes, important for device performance, were found, in our films, to be the highest reported. A thorough analysis of the optical response has shown that the normally expected energy gap signature will not occur in the new superconductors. A number of noncontact, nondestructive characterization techniques were applied including Raman spectroscopy, helium atom scattering (one of the most sensitive techniques for film and substrate surfaces), and newly developed eddy current homogeneity, surface degradation capacitance, and grating speckle whole field strain measurements.

Device related performance received its greatest effort in the final 18 months, having required the prior output of the synthesis and characterization programs. We have now produced a magnetometer which exhibits 50pT ($5 \times 10^{-7}G$) sensitivity, and is one of the first devices to utilize a bulk high T_c superconductor. This performance exceeds that of conventional magnetometers and would be adequate for magnetocardiograms. Weak links have now been produced by striped laser irradiation which provide rf detection response in the 10Hz-100Hz frequency range. Passive microwave performance of our laser ablated films measured by Sarnoff Labs has matched the best seen. Utilizing synchrotron sources, high T_c films were found to show a bolometric response which exceeds the speed of conventional detectors in some portions of the spectrum. A nondestructive method to switch a bulk superconductor from a high J_c state to a low J_c state has been discovered and a patent for these switching, storage, and erasure effects has been applied for.

Finally, a strong statewide effort in the theory of the new superconductors was also established. Considerable effort has been applied toward the solution of the Hubbard Model. Exact solutions in restricted cases and important advances in the more general problem have been achieved. Band structure and electron correlation effects have been calculated. Theoretical interest has ranged from

unconventional superconductivity with predicted new Josephson junction behavior, to equilibrium phase diagram calculations and the role of oxygen ordering.

Over 235 papers have been submitted or published, and nine patents have been applied for or allowed.

ADVANCED COMPOSITE MATERIALS

The Advanced Composite Materials program has produced significant advances in the processing, properties and characterization of composites for ultra high temperature applications. The overall objective of this program was to provide a fundamental understanding of the processing science and technology necessary to fabricate ceramic-matrix, intermetallic-matrix, and metal-matrix composites with superior mechanical properties in high temperature and oxidizing environments. The composites are intended for use as structural materials for advanced aerospace applications at temperatures exceeding 1200°C (2200°F). The program consisted of four interactive research projects in the areas of Fiber Fabrication, Coatings and Infiltration, Composite Fabrication, and Physical/Mechanical Properties. Significant accomplishments were achieved in each area. The programs have also led to extensive research collaboration with several industries, as well as other universities.

In the area of Fiber Fabrication, continuous silicon carbide fibers have been produced which have superior high temperature mechanical properties than other commercially available fibers. A patent application has been filed for this new technology. The most impressive is that the fibers have low oxygen impurities, because the fibers are produced without the oxidative crosslinking step, and are more stable at high temperatures. For example, the fibers show tensile strength around 1 GPa and 1400°C. In another project, short fibers of glass-free mullite with high relative density and fine grain size have been fabricated using sol-gel processing. The process has also been used to produce composite fibers containing tetragonal zirconia. The fibers show excellent microstructural stability after heat treatment in the range 1200-1500°C.

In the Composite Fabrication program, two new processes have been developed for producing intermetallic-based and ceramic-based composites. Patent disclosures have been filed for both. For the former, Reactive Hot Compaction (RHC) has been combined with an *in situ* coating technique to produce ductile metal reinforced aluminide matrix composites. The one step processing technique not only allows for the production of composites which are stable at high temperatures, but also allows for their economical processing. For the ceramic matrix composites, a process known as Transient Viscous Sintering (TVS) has been developed which involves viscous sintering and densification of submicrometer particles prior to the formation of crystalline compounds. The process has been used to produce high density, fine-grained mullite and mullite-based composites containing silicon carbide, silicon nitride and zirconia.

Microwave heating has been demonstrated as a viable technique for sintering and densification of ceramic matrix composites. Microwave energy has also been used to ignite self-propagating high temperature synthesis reactions in a variety of composites such as $\text{Al}_2\text{O}_3/\text{TiC}$, $\text{Al}_2\text{O}_3/\text{TiB}_2$, TiC/TiB_2 and $\text{Al}_2\text{O}_3/\text{SiC}$. In addition, a hybrid microwave and conventional heating technique has been used to attain accelerated densification and microstructural homogeneity in alumina-based composites.

In the area of Coatings and Infiltration, chemical vapor deposition was used to deposit TiC_x coating on a variety of substrates such as Al_2O_3 , SiO_2 , ZrO_2 , Ta, Nb and Nicalon. A dramatic reduction in

the deposition temperature has been achieved by utilizing the atomic layer deposition technique to produce this coating of controlled composition. The process involves sequential exposure of a substrate to TiCl_4 , then H_2 , then CH_4 , and then H_2 at temperatures as low as 500°C , compared with over 1000°C necessary for conventional CVD processes. In another project, a vinyllic polysilazene solution was developed which gives a high yield of $\text{SiC/Si}_3\text{N}_4$ ceramic upon pyrolysis. This material was used to produce adherent coatings on a variety of substrates, and for infiltration/densification of porous ceramics.

Various equipment and facilities have been installed which allow for processing and high temperature characterization of composites. The equipment include a high temperature mechanical testing facility, hot isostatic processing, high temperature dilatometer, high resolution digital scanning microscope, fiber spinning devices, etc. These facilities are used not only by the researchers in this program, but also by investigators from industries and other institutions.

Each of the four AMMP technology areas are summarized in greater detail in the following sections.

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Microelectronics

The primary objectives of the program were (1) to establish a capability for laser restructuring of wafer integrated circuits, (2) to examine the reliability of this technology, (3) to put in place the capability for researching alternative interconnect technologies and processes, (4) to explore the complex issues of design and test of large scale integrated systems, and (5) to examine system architectures which could fully realize the benefits of this technology. The results of this research include the establishment of a major capability to research technologies and architectures for future high performance systems - Ultra Large Scale Integration (ULSI) or Wafer Scale Integration (WSI) and the reporting of a number of significant technical accomplishments.

Laser Restructuring of Wafer Integrated Circuits

The Center for Microelectronics Research (CMR) Rapid Prototyping Laboratory was initiated in 1988 to support sponsored research by DARPA in Wafer Scale Integration. The original charter for this laboratory was to transfer the Lincoln Laboratory Laser Diffused Link technology to CMR for prototyping of wafer scale systems. While this transfer was successfully executed and the Laboratory is supporting this technology, other research activities have grown from this facility. The laboratory has greatly broadened its research scope to include state of the art processes in electronic interconnects for restructuring and rapid prototyping of wafer scale systems and hybrid multichip modules. Research being explored includes focused ion beam (FIB) depositions for submicron resolutions, laser direct writing of conductors, the maskless patterning of dielectrics for lift-off techniques, and laser created silicon vias for 3D wafer stacking.

The basic laser system consists of a vibrationless granite table with a high energy 5 watt Argon CW laser and a 65 watt excimer laser. The laser light is passed through the optics of the system and directed to a wafer chuck mounted on a high precision x-y-z positioning table (positioning accuracy is within 0.5 μm of the commanded position) with laser interferometer. This system is driven by a SUN workstation operating with elements of the SLASH (Standard Linking Automation SHell) software from Lincoln Laboratory. An IMS high speed tester is also a part of the system and allows dynamic verification of the breaking or making of interconnect links. The excimer laser allows tunable frequencies into the deep uv. The impact of a NdYAG laser is also being explored for incorporation into the lab. This will give the laboratory a wide spectrum of capability from the deep uv to the far infrared.

The Center for Microelectronics Research (CMR) has investigated various technologies for rapidly creating WSI products. These techniques would be used for restructuring defective areas as well as customization of generic elements. The predominate areas of research are currently with focused ion beams (FIB) and laser assisted processing. Laser processing includes direct writing of conductive lines, laser diffused links, and laser patterning for lift-off procedures.

Four universities within the state of Florida participated in an effort to develop unique WSI applications, as will be described later in this report. Configuration of the elements WSI will be accomplished by a technique developed at Lincoln Laboratories called Restructurable VLSI (RVLSI). These Alpha-Site universities will then be given WSI products for their beta site demonstrations. It is this use of a common processing element with laser restructuring that allows different applications to be quickly fabricated as a rapid prototype. The RVLSI technique involves lateral diffused links in which an argon laser beam hits between the two nearby diffused regions. The laser melts the silicon and allows the diffusion areas to merge, effectively creating a link.

These links have resistivity values on the order of several hundred ohms and therefore when high conductive links are needed several parallel links along the interconnecting lines must be made. This will impact the overall density capabilities of the design. Alternative laser or FIB links could allow higher device densities.

The same argon laser used for the lateral diffused link process was incorporated for the direct write of gold lines from a metalorganic compound. This process was relatively simple to perform with gold and further experiments will be undertaken to write with other metals. The basic methodology is to have the laser oxidize the organic constituent leaving the metal compound behind. For customizing WSI a method of opening vias to the desired regions for linking would be followed by the deposition of metal from this technique.

Focused ion beams have many favorable attributes, notably being able to achieve resolutions below 0.5 microns and able to etch and deposit materials in the same equipment. This tool appears similar to a scanning electron microscope except that heavy gallium ions are scanned for sputter etching and tungsten carbonyl is typically used for deposition. A test chip was fabricated by CMR with several different structures to evaluate links using focused ion beams. Potential link sites were arranged in a 10 x 10 grid with probe pads connected to each row and column. This arrangement permitted resistance measurements to be made on the FIB links. The type of links included: first level metal (M1) to second level metal (M2) lateral straps; M2-Poly lateral straps; M2-M2 lateral straps; M1-M2 vertical links; and 50 and 100 micron stripes for measuring sheet and contact resistance. Typical sheet resistances measured were 6.4 ohms per square and contact resistances were approximately 2 ohms.

Rapid prototyping for wafer scale integrated systems severely tax capabilities of existing software tools. Traditional hardware prototype configurations will yield little relevance for the advanced computing applications desired by going to a WSI technology. The preferred method is to attempt to rapidly produce final WSI products with the use of customizing techniques. These techniques can be employed for traditional restructuring for defect avoidance as well as allowing custom programmable devices. Therefore, utilizing programmable logic on a WSI system can slow diverse applications to be exercised. The Rapid Prototyping Laboratory has succeeded in exploring and developing alternative methods for the restructuring and rapid prototyping of wafer scale systems.

Interconnect Reliability

The objectives of this research task were (1) to develop and execute tests to determine the reliability of the diffused links which are the key to the Lincoln Labs Restructurable Wafer Scale Integration Technology, and (2) to develop destructive and non-destructive techniques and capabilities for the determination of integrated circuit interconnect reliability.

One potential major benefit to be realized from wafer scale integration is the significant increase in reliability to be realized from the drastic reduction in "off-chip" interconnects. Yet the transfer of interconnect technology from the board to the wafer presents its own challenges, not the least of which is understanding and assuring the reliability of the "on-silicon" interconnect technology to be used. There are two areas of focus for this investigation, (1) understanding the reliability (and potentially the manufacturability) of the specific interconnect elements, in this case the laser diffused links (LDL) and the laser cut fuses, and (2) developing general techniques for predicting the reliability of general interconnect technologies used in advanced integrated circuits. The first of

these is specific to the WSI or (ULSI) approach taken. For the case of the Lincoln Labs LDL Restructurable WSI technology, specific test structures and reliability tests are required to fully understand the long term reliability of the link technology.

Second is the development of non-destructive techniques for evaluation of current and next generation IC interconnect technologies. Interconnect technology is the major driver in increasing on-chip functional densities, whether by reducing interconnect pitch or adding layers of interconnect. Both of these approaches potentially degrade the overall reliability of circuit interconnections, by increasing current densities and process complexity. In addition to this is the trend to improve circuit performance by reducing interconnect resistance and capacitance through introduction of new interconnect metallurgies and multi-layer structures.

It is the potential of these new technologies and the "linking" techniques which are the major focus of advances in circuit integration, with expectations of corresponding improvement in system reliability - assuming that the new interconnects themselves do not present any new reliability problems, the research objective of this task.

Technical Accomplishments

Accomplishments included the following:

- An accelerated life test system for studying the reliability of laser diffused links and VLSI interconnects was designed, constructed and tested. The system can simultaneously stress eight samples at different current densities and temperatures.
- Three noise spectral analyzers, covering the frequency range from 0.1 Hz to 10 MHz were constructed, including an extremely sensitive dual channel spectral analyzer capable of detecting the thermal noise voltage of a 1Ω resistor at room temperature (or approx. 10^{-10} volts $\sqrt{\text{Hz}}$). The system has been used for reliability testing of Al-based interconnects with excess noise measurements.
- An automated noise measurement system for collecting the statistical noise data for reliability test analysis was designed, constructed and tested.
- Close correlation between the electromigration lifetime and the excess noise level of Al films have been found experimentally, and a physical model of the excess noise generation was developed to explain the experimental results.
- Our experimental results and theoretical analyses have shown that noise measurements can be used for reliability testing of Al-based techniques. This new technique is non-destructive and is more sensitive and much faster than conventional MTF measurements.
- Theoretical analysis was carried out to optimize the doping profile for laser-induced diode linking in WSI circuits. The results show that the laser linking resistance can be minimized by controlling the laser duration time.
- Preliminary reliability tests for laser induced diode linking were carried out for the samples obtained from Lincoln Laboratory.

A detailed summary of the results of the interconnect reliability task is contained in the AMMP Annual Report for the period 1 July 1988 through 30 September 1989 and in the referenced publications.

Process Technology

The research objective of the process technology task was to enable the investigation of interconnect strategies for advanced system development. A toolset was put in place which enabled the fabrication of both conventional interconnect structures as well as advanced structures. Included in this capability are thin film growth and deposition, pattern delineation, electrical and physical characterization instruments, and pattern transfer or etching systems for the thin films involved.

The initial research objective was to provide the capability of duplicating and testing the Lincoln Laboratories' Restructurable VLSI diffused link approach to wafer scale integration. The toolset associated with this structure involved thermal processing equipment, lithographic equipment, plasma and wet chemical pattern transfer systems, and various electrical and physical characterization systems.

To address the research goal of fabricating advanced interconnect structures, significant effort was expended to obtain quality tooling which could address the novel processing issues associated with such structures. Two examples of interaction with industry, which involved significant technical transfer, resulted in *first-of-a-kind* tooling which is now available and being purchased by domestic industry. These systems are the Electron Cyclotron Resonance (ECR) plasma emulator system and the Sputter Cluster Tool. The first tool was developed through faculty interaction with Plasma-Therm, Inc. of Vorhees, New Jersey, whereas the second resulted from interaction with Sputtered Films, Inc. of Santa Barbara, California. The ECR tool consists of a high vacuum emulator which facilitates advanced plasma source and thin film process development. The tool was initially fitted with a Wavemat compact ECR source. This disc source uses fixed rare earth magnets to generate the ECR plasma layer. With this source work was performed to develop low temperature thin film deposition processes for silicon dioxide and silicon nitride. Both materials were deposited at temperatures below 100°C and exhibited excellent physical and chemical characteristics. This tool was also used to study advanced etching processes associated with interconnect technologies. These included tri-layer resist processing, dielectric etching, and compound semiconductor etching. Faculty at USF have continued to work with Plasma-Therm, Inc., and the result has been follow-on funding to characterize, etch, and deposition process using new plasma ECR sources.

The interaction with Sputtered Films, Inc. resulted in the development of a new sputter cluster tool. Peter Clarke, the President of SFI, has extensive experience in sputter technology and is the inventor of the S-gun technology. Clarke and his development staff worked closely with CMR faculty to develop a tool which would facilitate the research and development of advanced interconnect structures, or other thin film processes. The result was a tool which embodies flexible process sequencing and open architecture for process development. The tool also leverages domestic R&D capability as its cost is roughly 25% of previously available cluster or integrated process systems. The cluster tool has been used extensively in CMR to investigate the reactive sputter deposition of silicon dioxide and silicon nitride, and to facilitate the development of 3-dimensional interconnect architectures. The tool is also responsible for the acquisition of follow-on funding to study refractory metal systems and structures to be utilized in circuits deployed in high temperature environments.

Plasma tooling obtained to deposit oxide and nitride thin films as well as etch circuit structures has also been used to study the high rate removal of silicon. Typical silicon removal rates are less than one micrometer ($1\mu\text{m}$) per minute. Using the plasma tooling available in CMR as the result of the AMMP program, silicon removal processes were developed which exhibit removal rates as high as $30\mu\text{m}^{-1}$. This work was performed in support of the three-dimensional WSI efforts at CMR. These results may also be utilized in other areas of silicon technology such as sensor fabrication, bond and etchback processes for dielectric isolated circuits, advanced packaging techniques, or mere wafer thinning for speed enhancement.

Silicon On Insulator (SOI) material development focused on improving the quality and reducing the cost of SIMOX (Separation by IMplanted OXYgen) wafers. A series of experiments was performed to evaluate the properties of SIMOX wafers formed by an experimental multiple oxygen implant/reduced oxygen implant dose procedure. Material quality was characterized by minority carrier lifetime, buried oxide defect density, buried oxide breakdown strength, and heavy metal contamination introduced by the implant. It was shown that good SIMOX material can be achieved with implant doses up to an order of magnitude below what is practiced today, thereby reducing wafer cost. An invention disclosure was submitted as a result of this effort. [Henley] Further work is required to understand the formation mechanisms and develop the process for commercial implementation.

The process capability established at CMR as the result of the AMMP program has supported other program tasks as well. Principal among these efforts have been those associated with the restructuring laser laboratory and three dimensional memory architecture and interconnect strategies. The toolset also was of major benefit to the contamination and defect engineering efforts.

Defect Engineering

A new defect engineering program has also been initiated at USF/CMR. A laboratory was established and equipped to measure defects and their electronics properties in silicon, SOI and III-V compounds. Equipment installed includes: Fourier IR absorption equipment, with Raman attachment for stress measurements; Deep Level Transient Spectroscopy (DLTS) for measurement of energy levels and concentrations of deep centers; scanning photoluminescence equipment allowing measurement for liquid helium (4.2 K) to room temperature and characterization of shallow acceptors, donors, recombination center, and mapping of impurity distribution; and Surface Photovoltage equipment allowing mapping diffusion length distribution of silicon or III-V samples. Rapid thermal annealing equipment was installed for contamination control experiments.

Initial research efforts have focused on the study of properties and characteristics of heavy metals in silicon, and the development of new non-destructive, non-contact characteristics techniques allowing measurements of heavy metals of the levels required by the microelectronic industry. As a result of these efforts, a potentially very significant new method for non-destructive, non-contact measurement of iron concentration in silicon with sensitivity of 1 part-per-trillion has been developed. This technique allows characterization of wafers at various stages of IC processing. The results achieved to date could provide significant cost savings to industry in terms of increased yield. [Lagowski, et al.]

WSI Applications, Architectures, Design and Test

Task objectives were: a) research on applications requiring massively parallel computations, and b) development of WSI architectures for implementation of these applications. The approach taken was two-fold: intensive study of the literature on Wafer Scale Integration, and concentrated research on carefully selected applications. Some key conclusions arising from the studies are the following: Architectures for WSI should have structural regularity (which requires the use of a single or very few species of cells), communication locality, and redundancy for defect tolerance. An integral part of the objective, therefore, is a critical study and assessment of reconfiguration methods/algorithms, i.e. the extraction of a functional wafer from a physical wafer with good and bad resources - cells and interconnect.

The objective of the design and test tasks was to develop and implement effective design and test methodologies for wafer scale and ultra large scale integrated system design. This means development of a closely coupled hierarchical design system which allows system designers to go from architectural concept and system requirements to a low risk, optimized physical WSI design in a timely manner. Specific elements which were targeted are the installation of a set of comprehensive design and test tools for wafer scale assemblies, and a partitioning of the design methodology, such that system designers can explore WSI architectural implementations at a high level.

Alpha-Site Project - WSI applications were developed at three Florida Universities (alpha sites): the University of Central Florida (UCF), Florida Institute of Technology (FIT), and Florida Atlantic University (FAU). A common wafer level design was defined which can be rapidly restructured to meet the system requirements of each university. A system design verification approach which involved defining a common CAD tool base for high level modeling was agreed upon by the multi-university team. This allowed each alpha site to develop high level models for their architecture using VHDL (VHSIC Hardware Description Language). The physical wafer structure can be automatically mapped to the wafer from the VHDL model port assignment definitions. Architecture, modeling, cell design, wafer layout, and WSI system development support for this project are being provided by the Center for Microelectronics Research (CMR) at the University of South Florida (USF).

In order to meet the signal and data processing requirements for a wide range of applications, a common WSI Processing Element (PE) cell architecture was jointly developed by CMR and the three alpha sites. [Landis, Brown, et al.] and [Landis, Athan, et al.] The cell is essentially a small programmable special purpose CPU tailored toward parallel signal or data processing and neural network applications. The cell has 40 signal inputs, 17 signal outputs, and 2 bi-directional I/O signals.

An instruction set definition was developed by CMR for the alpha-site PE cell based upon requirement inputs from each of the alpha sites. In order to minimize the number of global (wafer level) control lines, an encoded set of control lines are decoded by the PE cell, corresponding to this pre-defined instruction set. The VHDL cell model and the cell physical design were developed at USF/CMR. The alpha sites can ultimately incorporate the detailed cell level VHDL model with their complete architectural level VHDL simulation activities. Final system level verification can be performed for all three architectures prior to submitting the wafer design to fabrication. In

addition, FIT has developed a software translation tool which will create the required WSI system netlist directly from the VHDL architectural model. This tool automatically creates the input file to the MIT Lincoln Labs SLASH tools, which are then used for automated WSI reconfiguration.

Successful test results for both the complex multiplier and STI were reported. [Singh] A modified version of the STI was created in Cadence for use in the alpha-site and PSDP wafer designs.

Physical design of this wafer was performed at CMR on Cadence using 2 micron CMOS rules from MOSIS so that wafer fabrication will not be locked in to any single foundry. The layout of the test chip for the PE cell measures approximately 3 mm on a side. The data inputs to the PE are located at the top of the cell and the data outputs are located at the bottom. This organization will simplify the data path interconnections between PEs. The actual wafer tile structure will contain both true and mirrored versions of this cell, in adjacent columns.

The neural network design developed by UCF and Martin Marietta in Orlando as their WSI alpha-site application consists of a three-layer network, with each layer containing a maximum of 32 neural network processing elements. This architecture is oriented toward small field target recognition applications which can benefit from the rapid prototyping capability and high density availability using RVLSI wafer scale technology.

FAU teamed with IBM Entry Systems Division in Boca Raton has developed a wafer scale architecture of an image perception system based on a biologically influenced computational paradigm (ALOPEX). ALOPEX uses a stochastic procedure to find the global optimum of linear and nonlinear functions. Using the Boltzmann probability distribution function, the ALOPEX algorithm generates probabilities of taking positive or negative steps away from its current position on the path toward the global optimal solution. [R. Shankar]

The Processing Element cell of our alpha-site WSI design is well suited for implementing a wide variety of digital signal processing algorithms. The single cycle parallel multiplier/accumulator will support high speed digital filtering and FFT operations, and the programmable ALU adds flexibility for application customization. FIT has developed a novel architecture which will exploit many of the features of our WSI PE and rapid prototype wafer system architecture and has teamed with Harris (Melbourne, Florida) as their industrial partner.

Programmable Systolic Data Processor Project - The Programmable Systolic Data Processor (PSDP) is being designed to enhance DoD mission capabilities by extending signal and data processing speed/performance while reducing system size, weight, and power consumption. Programmable fine-grain multicomputer concepts with distributed memory have been explored at Honeywell, Inc. This work has been extended at the Center for Microelectronics Research of the University of South Florida under DARPA support through the conceptual development of the PSDP. The characteristics of the PSDP architecture: broad homogeneity, ease of redundancy, and limited physical interconnect requirements make it opportune for building as a wafer scale system. The potential for exploitation of ultra-large scale integration with high interconnect bandwidth, using a robust programmable systolic array processing architecture, provides unique on-board processing capabilities for DoD missions.

A key to the full exploitation of the benefits of WSI processor performance involves the physical interconnection and packaging of a multi-wafer system. Honeywell and CMR are working as a team to develop the PSDP architecture/design as well as a unique and advanced stacked WSI interconnect technology that intrinsically boosts system performance and reliability, while further lowering size, weight, and power consumption.

Unique features of the PSDP architecture include distributed control and software allowing each processor to act as a self-controlled node containing its own programming. The present PSDP architecture takes advantage of an orthogonal memory structure to allow 8 bits of data to be read concurrently with a 32-bit instruction.

Optimization of the processor architecture is required for planar wafer scale implementation. In addition to design optimization for defect tolerance and inter-cell connectivity, issues such as power minimization and enhanced testability are being addressed.

Under DARPA support, CMR has developed a VHDL model of the PSDP processor cell as well as a 10 node by 10 node architectural VHDL model. These models are being used to perform system level evaluation and trade-off studies. The VHDL cell model has been transferred to the Honeywell CAE system and is being used for high level system trade-off analysis.

A complete multicomputer system can be constructed using a single programmable systolic processing element building block. The homogeneity of this architecture maximizes the potential use of all functional cells of each wafer. The PSDP operates with nearest neighbor communication (including virtual nearest neighbors) making the design easy to harvest into a 2D mesh of nodes on a wafer. Furthermore, the low cell I/O count minimizes the wafer area required for processor-to-processor interconnections.

A key element in the vertical stacking of die or 3D Stacked Wafer Scale Integration (SWSI) involves the etching or drilling of holes through wafers to produce the necessary Z-axis vias. These holes must be insulated with a dielectric and then plated or filled with a conducting material. The technology being developed at CMR for 3D interconnection of chips and wafers can be used to produce a prototype stacked wafer scale PSDP system. This Z-axis interconnect capability provides fundamental system level advantages in that memory can be significantly expanded with very little increase in "distance" from the processor to the memory.

The complexities of test for wafer scale systems have been addressed at CMR with a four point approach: (1) development of a structured test methodology based on use of the IEEE 1149.1 (JTAG) Standard Test Interface, boundary scan, and built-in test; (2) incorporation of test structures in selected regions of the wafer to allow for wafer acceptance testing based on parametric measurements; (3) application of scan test at wafer level through probing of the 4-pin IEEE 1149.1 test access port; and (4) performance testing of the restructured wafer scale system using functional and manufacturing test patterns.

A Standard Test Interface (STI) for WSI has been developed at the University of South Florida, based on the IEEE 1149.1 test bus standard. [Landis] and [Landis, Nienhaus, and Nigam] It consists of an 1149.1 Test Access Port along with serial pattern generator and signature analyzer circuits to facilitate Built-In Self-Test. An STI prototype was fabricated by MOSIS and tested at CMR as part of an FFT complex multiplier test chip.

High Speed Digital Test Development - The objective of this task is to develop the capability to test digital logic devices at data rates in excess of 1 Gbps. Initially we have set an objective of reaching 1.5 Gbps during 1990. This initial goal was attained with the development of an 8:1, digital GaAs multiplexer which was connected to an HP82000 200 MHz test system. The resulting data rate was 1.6 Gbps. In addition to the primary objective of providing high data rate patterns for testing, we have set specific performance objectives relating to the flexibility of the developed test system. These include (1) the ability to handle multiple (8 to 16) high speed signals

in conjunction with the synchronous operation of as many as 120 moderate frequency signals (up to 200 MHz); (2) the ability to program timing edges with 50 ps resolution and comparable accuracy; (3) a "tester per pin" architecture which provides the ability to independently program the timing, format, and logic levels of each signal; (4) support a pattern depth of at least 64k per pin.

The approach we have taken involves leveraging the capabilities of a commercial test system (the HP82000) by adding digital GaAs logic and analog components to the test head thereby supporting the multiplexing of several (either groups of 4 or 8) 200 MHz channels to generate signals in the range of 800 Mbps to 1.6 Gbps. Initial results were obtained using off-the-shelf 100k ECL components to perform 8-to-1 multiplexing at rates up to 800 Mbps. Recently we have built circuits using digital GaAs which perform 8:1 multiplexing up to 1.6 Gbps. An analog "pin driver" circuit was added to this arrangement to permit the programming of logic levels on a "per pin" basis. This device generated logic transitions in 250 ps with amplitudes up to 6V. The driver limited data rates to 1.2 Gbps. However, with the driver bypassed, we were able to achieve the full 1.6 Gbps rates using the GaAs logic levels. We were also able to achieve rates up to 3.3 Gbps in short bursts of 8 bits, followed by 2ns "dead" time during which the test pattern data is held at its prior state.

The Microelectronics Test Laboratory includes the HP82000 high speed digital test system, a Thermonics temperature forcing system, a high speed (1 GHz) oscilloscope (Tektronix 11403), and a SUN 3/60 workstation used to run the Cadence design and simulation software. Recent capability improvements have included the addition of (1) a 256 signal, 50 Ohm coaxial wafer probe interface from Cerprobe; (2) a Thermonics T-2420 temperature forcing system for -65 to +200°C operation; (3) expansion of the HP82000 pincount from 152 to 200 channels; (4) a 1 Gbyte hard disk drive; (5) test pattern translation software from TSSI. The characteristics of the HP82000 test system include: (1) 200 MHz clock and data rates; (2) 200 bidirectional test channels, expandable to 384; (3) +/- 250 ps timing accuracy; (4) 50 ps timing resolution; (5) subnanosecond edge rates; (6) a "per pin" architecture; (7) a complete DC characterization capability.

The system architecture of the Ultra High Speed Test System involves logically segmenting the HP82000 test head so that a Device Under Test (DUT) performance board can provide direct interconnect between the DUT and the HP82000 on the right side of the system. The left side provides generation of ultra high frequency data by using GaAs modules to multiplex groups of 4 or 8 test signals. The resulting 800-1600 Mb/s signals are connected to the DUT via an arrangement of SMA or SMB connectors and coaxial cable.

The logic used to perform high frequency multiplexing is relatively simple, requiring an arrangement of off-the-shelf GaAs exclusive-Or gates connected to form a multiple input XOR tree. The output is connected to a GaAs "pin driver" which allows the high and low logic levels to be independently programmed. The XOR tree requires that the input signals be encoded and phase delayed. The ability of the HP82000 to precisely control the timing of each signal with 50 ps resolution is key to the success of this approach.

We have successfully demonstrated generation of 1.6 Gbps data patterns using the prototype 8:1 multiplexer. This has included the equivalent of an 800 MHz clock and its complement and arbitrary 1.6 Gbps data patterns. The developed system includes the ability to program delays in a given signal with 50 ps resolution and approximately ± 100 ps accuracy. Examples of the pin driver output illustrate rising and falling edge transitions (20-80%) in less than 250 ps. The pin driver ability to adjust both the high and low logic level voltages through a 6.0V range has been demonstrated.

To establish the upper limit of operating frequency for the GaAs logic, a short burst of 8 bits are applied to the 8:1 multiplexer. The resulting output signals have an effect rate of 3.3 Gbps. This represents the upper limit of operation for the devices and PCB used in the prototype circuit.

We have worked closely with Honeywell Corporation in developing tests for a 272 pin tab-leaded VHSIC device. A 5-layer printed circuit board was designed to form a custom interface between the device and the HP82000 and translated test patterns from the Honeywell "QuickSim" format to the HP82000 using the HP EDA software.

Hewlett Packard's interest in the test development activities at the CMR include: (1) the extensions of test capabilities above 400 MHz; (2) joint development of high speed test applications notes; (3) organization of the last two HP82000 International User's Group Meeting - in June 1990 the CMR hosted the meeting of over 70 test engineers from around the world, and this two day workshop was conducted again at USF on April 4 and 5, 1991; (4) development of a standard parts test library for TTL, CMOS, and ECL devices; (5) development of a tutorial on "High Speed Testing with the HP82000" which was presented at USF on April 3, 1991.

Optoelectronics

A number of world records have been set and milestone achievements reported by the faculty participating in the Optoelectronics section of Advanced Microelectronics and Materials Program. These are discussed below in sections describing progress in compound semiconductors growth, processing and devices; thin film optical materials and devices; laser host and nonlinear optical crystal growth; and high resolution, high brightness displays.

Compound Semiconductors

In the area of compound semiconductors, the objectives of the program were to develop a Center of Excellence for electrically injected compound semiconductor visible emitters which will enable higher density optical storage, high resolution and brightness displays and provide proper wavelengths for compact and efficient diode pumping of solid state lasers. Further, the quantum well devices being produced and tested are critical to OEIC's, for faster and low noise devices and for higher power levels.

For II-VI semiconductors, MBE has been used to reproducibly grow p-type ZnSe with world record doping densities of up to $8 \times 10^{17} \text{ cm}^{-3}$. [Park] To achieve this level of doping, plasma cracking of N_2 was used in a novel approach. World record low resistivity of these epilayers was measured using a contactless method to be $0.75 \Omega\text{-cm}$, and the photoluminescence spectrum and C-V data agreed that the material was p-type. Ohmic contacts to these p-ZnSe have been demonstrated, however their resistance is very high. [Holloway] ZnSe layers doped n and p-type were heteroepitaxed onto GaAs substrates with the p-layer buried under an n-ZnSe surface layer. Ohmic contacts were made to the front n-ZnSe with indium and to the back p-GaAs with gold, and a p-n junction ZnSe LED was manufactured with an emission centered at 460 nm. [Park] This world's first technology for reproducibly growing p-ZnSe has been transferred to 3M Co. who successfully demonstrated an electrically injected 470nm diode laser operating at 77K, and a green diode laser operating at room temperature. In addition, p-doping technology has been transferred to a number of laboratories around the US including Phillips Research, North Carolina State University, and Purdue University.

Growth of ZnSe by MOCVD has been accomplished and novel precursors for nitrogen doping have been demonstrated to result in incorporation of N in the lattice. [Anderson] Attempts to ion implant and rapid thermal anneal the ZnSe for doping were successful in developing overpressure techniques to avoid point defect generation during annealing, however the implanted species were not electrically active. [Jones] Infrared optical constants and a new analysis technique have been used to characterize the carrier concentration in ZnSe by a method which avoids problems with ohmic electrical contacts. [Deneuve, Tanner and Holloway] In addition, demonstration of room temperature photo-pumped lasing in ZnSe bulk crystals and epitaxial layers, using a new resonance technique, was demonstrated for the first time. [Zory]

For III-V semiconductors, the variability of dopant concentration in bulk GaAs crystals grown by the Czochralski technique was assessed. [Witt] A novel scheme for the MBE growth of device quality InGaAs heteroepitaxed to lattice mismatched GaAs substrates has been demonstrated using a superlattice of AlGaAs/GaAs in combination with buffer layers of InGaAs to blunt propagation of dislocations from the heterointerface into the active InGaAs layer. TEM analysis of the material showed that the active layer had a dislocation density of $<10^5 \text{ cm}^{-2}$. [Park] P-I-N photodiodes

operating at $1.3\mu\text{m}$ were fabricated from these epilayers and the dark current measured to test the quality of the material; very low dark currents of $\approx 10^{-6} \mu\text{A}$ were measured, indicating that the material was of very high quality. [Park and Li] This technique should make InGaAs quantum well diode lasers possible on GaAs substrates to facilitate integration of OEIC's.

Epitaxial layers of III-V materials were grown by MOCVD as well as by MBE. In a significant step forward, we addressed the safety issue of this growth by demonstrating that elemental sources could be used to grow device quality GaAs and InP based alloys. [T. Chu and S. Chu] In addition, a large bandgap ternary alloy of InGaP has been grown by MOCVD in the double heterostructure configuration for LED's with wavelengths of 580 nm and 603 nm. [Anderson] This material was used to fabricate edge-emitting LED's at an industrial partner, EPITAXX, giving orange light emission. [Anderson] Patterned area growth has been studied in the III-V and II-VI systems to reduce interfacial dislocation densities [Anderson] and to enhance the ability to integrate for OEIC's. [Das] Selected area growth of InGaAsP on InP has been demonstrated for the first time. [Anderson] Ion implantation and reactive ion etching damage in the III-V has been explored to develop new processing techniques and limits. [Jones] In addition, ion implantation interdiffusion effects from focused ion beam irradiation was used to produce a lateral modulation of the band gap in GaAs-AlGaAs structures. [Petroff] A new mechanism for formation of ohmic contacts to GaAs has been demonstrated through segregation of dopants during decomposition of the substrate by the contact metallization. [Holloway and Powell] New and improved characterization techniques have been developed using Raman [Anderson, Hummel and Sundaram], luminescence and reflectance [Stillman, Sundaram, and Hummel], SIMS and resonant ionization mass spectrometry [Eyler] and photon-enhanced impedance spectroscopy. [Orazem] A new technique called Frequency-Domain Photoluminescence has been developed to measure the bulk lifetime and interface recombination velocities of GaAs. [Neugroschel] This technique was used to show that the free sulfur-buffered solution developed to sulfur passivate GaAs surfaces was a significant improvement over previous methods. [Holloway]

Milestone achievements were reported for a MQW/SL AlGaAs/GaAs grating coupled photodetector with low noise (dark current $< 10^{-5} \text{ A}$ at 77K), high speed ($> 1\text{GHz}$) response at $10 \mu\text{m}$ and $D^* = 10^{10} (\text{cm}^2\text{-Hz})^{0.5}/\text{W}$. [Li] An ultra-high gain, low noise AlGaAs/GaAs Separate Absorption Multiplication (SAM) Avalanche Photodiode (APD) with a QE of 70%, peak response of 0.45 A/W at $0.8 \mu\text{m}$, a 3 dB cutoff at 1 GHz, and maximum internal photocurrent gain of 6040 with a breakdown voltage of 90V was demonstrated. [Li] A novel planar dual wavelength InAlAs/InGaAs photodetector was demonstrated using graded superlattices. Responsivities of 0.34 A/W and 0.42 A/W were demonstrated at 0.8 and $1.3 \mu\text{m}$, respectively, with a 3 dB cutoff at 10 GHz and 45GHz, respectively. [Li] The $1.3\mu\text{m}$ device represents a world record in terms of the responsivity-bandwidth product. The first successful fabrication of a high performance planar InGaAs photodetector heteroepitaxed to a GaAs substrate and operating at $1.3\mu\text{m}$ was demonstrated using a novel strain relief method. [Park and Li] Device physics models of heterojunction bipolar III-V transistors have provided improved SPICE models for small-signal, high frequency inputs, as well as large-signal nonlinear transient inputs. Specific accomplishments include development of the first accurate model of emitter-base heterojunction space-charge-layer capacitance and recombination currents, development of the first heterojunction bipolar transistor models that include non-quasi-static effects due to electron and hole velocities, development of the first model for base pushout effects, and inclusion of arbitrary doping and energy-gap profiles. [Lindholm and Liou] Finally, the effects of strain on nonlinear absorption and carrier dynamics in GaAs over femtosecond times has been explored using theoretical models. [Stanton]

In terms of OEIC's, significant progress was made in using III-V materials for waveguides, switches and modulators. Ridge waveguides of GaAs on Si with losses of $< 1 \text{ dB/cm}$ and

modulation efficiencies of 4.7°/mm-V were demonstrated. [Ramaswamy] A novel, high performance P-p-i-n-N GaAs/AlGaAs DH waveguide phase modulator with phase-shift efficiencies of 82°/mm-V at 1.06 μm with a bandwidth of 4.2 GHz was developed. [Ramaswamy and Srivastava] All-optical switching over 250 μm lengths in GaAs/AlGaAs MQW's with 100 psec recovery times (with carrier sweep-out) was demonstrated on twin stripe couplers on GaAs. [Miller] Cross-well carrier sweep-out on GaAs/AlGaAs p-i-n SEED devices improved the recovery times to <10 psec, and the effect interpreted using theoretical models of SEED devices. [Miller] Optical switching with gain was demonstrated in an InGaAs/GaAs MQW laser biased just below threshold. [Park and Miller] OEIC's have been incorporated into an image-rejecting, coherent detection system using a unique subcarrier modulation scheme that allowed demonstration of 600MB/S data rates with <10⁻⁹ error. The MMIC's and OEIC's developed were tested in the system. [Lachs, Henning and Bhattacharya]

Thin Film NLO Optical Materials

Thin film optical materials are critically to successfully fabrication of OEIC's and interconnections for optical signal processing. New materials and processing technologies have been demonstrated in the program, including sol gel processing of quantum dot and waveguide materials. The highest quantum confinement shift and NLO response reported to date for <10 nm compound semiconductors crystallites in a glass matrix has been demonstrated. [Simmons] The third order nonlinearity of GeO₂-SiO₂ and TiO₂-GeO₂ glasses were measured and the nonlinearities of glasses containing TiO₂ were twenty times larger than those containing SiO₂. [Stegeman and Simmons] Productions of single phase KNbO₃ thin films using bimetallic alkoxides precursors and sol gel processing has been accomplished for the first time. These films exhibited preferred orientations on KBr and SrTiO₃ substrates. [Sacks] It was further demonstrated that microwave processing of this sol gel results in faster dehydration and organic binder burnout. Microwave processing was used to produce single phase KNbO₃ thin films. [Clark] Deposition of SiC films from a plasma has been demonstrated [Buckle and Ammons], as has improved optical coatings with more uniform, higher values of dielectric constant using the Reactive Low Voltage Ion Plating (RLVIP) techniques. [Guenther] The technology of RLVIP was transferred to Martin Marietta Optical Components Center, Orlando. Ion implantation was used to produce a cladding region for optical fibers. [Djeu] Thin films of AlN and GaN epitaxed onto sapphire substrates were grown using trimethylaluminum and nitrogen trifluoride. [Edgar] Finally, silver/sodium exchange in BK7 glass was used to fabricate symmetric directional couplers. MUX/DEMUX operation at 1.315 and 1.561 μm with a crosstalk and cross power ratio of <-40 dB were demonstrated with insertion losses of <1.25 dB for 14.5 mm devices. [Srivastava and Ramaswamy] Waveguides in LiNbO₃ were defined by proton-exchange and Ti diffused for a quasi-phase matching condition which allowed conversion of a laser output from 860 nm to 430 nm with an efficiency of 65%/W-cm². [Srivastava and Ramaswamy]

Crystal Growth

In the area of laser host and nonlinear optical crystal growth, the AMMP effort is intended to develop a US Center of Excellence in the intelligent growth of bulk crystals with improved solid state laser inversion lifetimes, tunability, conversion efficiency and power output, as well as improve the conversion efficiency and power handling capabilities for frequency doubling and tripling. Towards this end, a state-of-the-art facility using expert systems for intelligent high temperature Czochralski growth of a number of fluoride and oxide crystals has been established. [Shah; Chai] The facility consists of 5 Czochralski pullers, 3 flux growth furnaces and 2 liquid

phase epitaxy furnaces. One Czochralski puller uses an OPS83 expert system and Computer Vision techniques. These furnaces have been used to grow a number of new crystals, including chromium doped lithium strontium aluminum fluoride (Cr:LiSAF) which exhibits lifetimes ($\approx 70\mu\text{s}$) sufficiently long to allow flash-lamp or laser diode pumping of the material. [Stalder, Bass and Dixon] Cr doping to above 10% has been achieved and shown to result in high conversion efficiency in a laser. [Stalder and Bass] The output has been shown to be tunable with good power output from 780 nm to beyond 1040 nm and maximum power out at 860 nm. At this wavelength, a slope efficiency of 7.1% and maximum overall efficiency of 5.5% with an average power output of 10W was demonstrated. [Stalder and Bass] The material was also ran in the Q-switched mode and a maximum slope efficiencies of 1.6% at 2Hz and 150 mJ output in 45 nsec was observed to be the damage limit. [Stalder, Bass and Soileau] Mode locking produced 500fsec pulses at 80MHz when pumped with a cw krypton laser. [Miller] The 10%Cr:LiSAF was also cw diode pumped with an AlGaInP diode laser at 670 nm, and a threshold power of 3 mW was determined. [Dixon] Continuous tuning was possible from 835 nm to 890 nm when the LiSAF was pumped by the 30 mW output of a 670 nm dye laser. [Dixon] The output of the Cr:LiSAF was doubled and 5 mW of power was produced at 435 nm in the blue region. The crystal growth technology for LiSAF was transferred to Lightning Optical in Tarpon Springs, FL, who also licensed the rights to growth from Lawrence Livermore National Laboratory. Thus we have created a US supplier of this extremely promising material.

In addition to LiSAF, significant improvements in the procedures for growth of rare earth doped yttrium lithium fluoride (YLF) crystals has been achieved (doped with Pr, Nd, Er, Ho, Tm, and Yb). [Chai] The new growth procedure considerably simplifies the seeding and pulling of these crystals, reduces wastage due to cracking and improves the product yield. [Chai] This technology was also successfully transferred to Lightning Optical in Tarpon Springs, Florida, which is now producing commercial crystals for the US market.

Finally, a number of other crystal have been grown in this facility, including bismuth strontium oxide (BSO), barium borate (BBO), lithium borate (LBO), lithium calcium aluminum fluoride (LiCAF), lithium sulfur gallium fluoride (LiSGaF) and lithium boron aluminum fluoride (LiBAF). [Chai] Solution growth facilities have also been established and a number of crystal grown using this technique for frequency doubling and tripling.

High Resolution, High Brightness Displays

Efforts over the last year resulted in significant progress towards devices and system requirements for high brightness, high resolution displays. High efficiency frequency doubling of YAG using double pass harmonic generation to produce green wavelengths was demonstrated. [Dixon] Thin film transistors of CuInSe_2 were produced for active matrix thin film displays. [Morel] Operation of an acousto-optic laser imager consisting of a low-power solid-state laser, optics system, galvanometer, and drive circuit for the acoustic crystal was demonstrated. [Glenn]

Summary

Benchmark achievements have been reported in the intelligent growth of crystals for solid state lasers, and for the growth and processing of LED's, diode lasers, and photodetectors from ZnSe and III-V compound semiconductors.

Superconductivity

The following is a summary of the accomplishments of the Superconductivity Program with the DARPA sponsored Advanced Microelectronics and Materials Program. These accomplishments are divided within the four principal themes i) Synthesis and Processing, ii) Characterization and Properties, iii) Device Related Phenomena, and iv) Theory. More than 235 papers describing these findings have been published, accepted by, or submitted to scientific journals (see Superconductivity References) and 9 patents have been applied for or allowed. Approximately 200 oral presentations and "papers in progress" have also resulted.

Synthesis and Processing

A new, simple, nonvacuum environment method to produce patterned films onto transparent substrates in one step has been developed. Synthesis of various films has been achieved including magnetic materials and superconductors using this back-transferred laser ablation technique. A patent application has been made. [Hascicek and Kleinhammes]

New thin film substrate materials in the form of single crystals of SrLaAlO_4 and CaLaAlO_4 have been grown. [Chai] The dielectric constant from 4.2K to 300K at frequencies from 20Hz to 1MHz has been measured. [Jenks]

A thin film non-line-of-sight laser ablation technique has been developed and shown to improve surface smoothness more than the use of shorter laser wavelengths. [Chang, Kennedy] Transport critical currents of 4×10^6 Amps/cm² at 77K and 2×10^7 Amps/cm² at 4.2K have been obtained for the non-line-of-sight laser ablated films. These are at the highest values reported for the materials. [Hascicek]

Studies of sputter deposited film composition and uniformity showed that starting with BaO_2 rather than BaCO_3 resulted in much better films. Use of BaF_2 resulted in even better reproducibility. [Leskela, Mueller, Truman, Holloway]

It was shown conclusively for the first time that the cause of sputtered film nonuniformity in composition and thickness resulted from negative oxygen ion resputtering and the long time to achieve constant film composition. The maximum ion flux came from the orthorhombic superconducting phase, suggesting that nonsuperconducting, non-consolidated targets were best for controlled deposition of thin films. [Leskela, Mueller, Truman, Holloway]

Barrier layers of metals (Ag and Cu) were deposited and shown to be ineffective at avoiding interdiffusion, but effective at maintaining the superconducting state. Barrier layers of SrTiO_3 , MgO , and Al_2O_3 were deposited and shown to be somewhat effective at limiting interdiffusion. Auger analysis showed that SrTiO_3 does not eliminate interdiffusion, but the substitutions have very little effect on the superconducting transition temperature. Both MgO and Al_2O_3 limited the extent of interdiffusion; however, the transition temperatures were lower. [Leskela, Mueller, Truman, Holloway]

Several new barrier layer materials were identified as promising to provide lattice matching to develop texture, good stability to limit interdiffusion, and with a low dielectric loss coefficient at

microwave frequencies. Tests to date suggest that some of the chromates may indeed make good barrier layers. [Leskela, Mueller, Truman, Holloway]

Films of $\text{YBa}_2\text{Cu}_3\text{O}_7$ have been deposited on LaAlO_3 substrates up to two inches in diameter with $T_c > 88\text{K}$ ($R = 0$, $\Delta < 3\text{K}$), $J_c > 10^4$ ($H = 0$, 77K), and thickness uniformity (0.25μ coating) of $< \pm 2\%$. The compositional nonuniformities are $< \pm 1\%$ for these crystalline, textured (preferentially oriented c-axis perpendicular to substrate surface) films. Growth has been accomplished by plasma enhanced organometallic chemical vapor deposition at substrate temperatures $< 700^\circ\text{C}$ to yield in-situ film properties not requiring a post-synthesis annealing. Numerous new source compounds for the traditionally involatile group 2 elements have been discovered. Bubbler use temperatures have been reduced to $< 200^\circ\text{C}$, eliminating the need for high temperature valves. Three new FSU compounds currently are being used by other thin film growers in an industrial collaboration. [Rees]

Diffusion barrier structures of Cu/Mo/Si and $\text{Cu/MoN}_x/\text{Si}$ were found to be effective in preventing the Cu from reaching the Si where its presence would result in deep level traps. [Burns]

Laser ablation of $\text{YBa}_2\text{Cu}_3\text{O}_7/\text{PrBa}_2\text{Cu}_3\text{O}_7$ modulated structures with x-ray sideband determined wavelengths down to two unit cells (24\AA) has been achieved with $T_c > 60\text{K}$. These superconducting-nonsuperconducting microstructures, synthesized in just four laboratories in the world, will be used in electric charging, weak link, and superconducting electrode capacitor device studies. [Kennedy]

Free standing laser ablated thin films of 123 with transverse dimensions up to 6mm have been obtained. Modelling studies of their bolometric performance, which could be enhanced, have been made. These are the only reported free standing films of a high T_c superconductor. (Patent applied for). [Ng]

A modified flux technique for the growth of free standing single crystals of RBaCuO ($R = \text{Y, Ho, Pr}$) with linear dimension up to 6mm and the provision of low resistance contacts thereto, was developed. [Gielisse] Good quality $\text{YBa}_2\text{Cu}_3\text{O}_7$ and $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_8$ crystals were also grown and characterized at a separate site. [Stewart]

Superconducting samples of $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ were produced using screen-printing, tape-casting, pressing, and slip-casting techniques. Monoliths of 123 were prepared by self-propagating high-temperature synthesis (SHS) in the microwave oven. (Patent to be granted). [D. Clark]

Silver and silver oxide were added to bulk and screened 123 in quantities up to 15 volume percent, which improved environmental stability and mechanical durability of the superconductors. [Gielisse, D. Clark]

An excellent preparation procedure for the thallium compound which reduces the thallium loss, allows equilibration with oxygen, and permits the loss of absorbed water has now been developed. [R. Clark, Nicolesceau]

We have developed an oxalate method for preparing the rare earth 123 superconductor that has lead to excellent quality material as pure single phases, as mixed rare earths, and as material deliberately off composition or impure. [R. Clark, W. Wallace]

Improvements in the size and orientation of microcrystals in high T_c superconductors through refinement processing have been achieved. [Leventouri]

Single crystals of $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_8$ were electrochemically doped with lithium ions at room temperature, resulting in a first-order crystalline/superconducting to amorphous/insulating phase transition. The diffusion coefficient for Li ions is slow enough (ca. $10^{-11} \text{ cm}^2 \text{ sec}^{-1}$) to allow extremely thin insulating layers to be introduced on the surface of the superconductor. This layer is reactive and can be derivatized further (e.g. metal or polymer boundaries can be deposited). A route to barrier layers or weak link fabrication may be opened up. [Schlenoff]

Characterization and Properties

Persistent current lifetime measurements, relevant to thin film device performance, show an approximate ln time behavior with <2% decay in the first 24 hours at 5K after 100 Oe field cooled induction. These results, obtained on our non line-of-sight laser ablated films, may be the best reported performance. [Kleinhammes]

Systematic Raman microprobe and FTIR studies of $\text{YBa}(\text{CuM})\text{O}$ with $\text{M}=\text{Fe}, \text{Co}, \text{Ni}, \text{and Zn}$ with oxygen variation, and with environmental degradation have been carried out. The complex dielectric function from 4000\AA - 8500\AA has been measured by ellipsometric techniques for 123 films. [S. Sundaram]

It was shown definitively that high T_c materials are in the clean limit for superconductivity, making the superconducting energy gap difficult to see by infrared spectroscopy, especially because there is a strong non-Drude absorption in the midinfrared region. This mid-IR absorption masks any weak effects due to the superconducting gap. It was also shown that the infrared spectroscopy could measure the mean free time between collisions of the charge carriers. The analysis gave a weak-coupling result for the interaction of these carriers with thermal excitations, such as phonons. Finally, it was shown that nearly all of the free carriers condense to form a superfluid below the superconducting transition. [Tanner]

Ac calorimeter techniques were developed to measure specific heat of <1 mg samples of high T_c superconductors up to 120K. Among the results is the accurate (~ %) determination of the oxygen content in superconducting La_2CuO_x . [Stewart]

Environmental surface degradation has been studied by electron microscopy [Desai] and rate determinations made by capacitance measurements. [Larkins] Environmental stability of 123 superconductors was found to be affected by processing parameters. The annealing cycle was found to be extremely critical in determining the quality of superconductors, including resistance to degradation. Slow cycling between 950°C and 400°C under flowing oxygen gave the best results. High compaction pressures including hot pressing did not improve surface degradation characteristics, even in >99% dense specimens. Neither polyimide or epoxy coatings were found suitable as protective coatings. The tests also showed that the BaCO_3 film which forms upon environmental exposure is initially non-protective. [Desai]

Helium atom scattering was used as a new tool to probe the surface structure and dynamics of the 123 and 2212 forms of the high temperature superconductors and to examine the surface substrates used in the preparation of laser ablated thin superconducting films. Substrate surfaces were found to be very rough on the atomic scale for all surfaces examined which were in the form of a cut and

optically polished surface. Cleaving a substrate in air (such as MgO) improved the surface quality and cleaving in vacuum greatly improved the surface quality. [Skofronick]

A new noncontact, nondestructive eddy current method to determine the electrical homogeneity of bulk materials has been developed. The method clearly demonstrates the large spatial inhomogeneity which results from normal bulk powder processing and its variation resulting from heat treatment and provides a good characterization tool for bulk materials. [Jenks]

A new method for detecting orientation in bulk samples based on polarized Raman spectroscopy has been developed. [Leventouri, Medina]

A high sensitivity, Grating Objective Speckle Method, which combines speckle metrology and a grating technique, has been developed for inplane displacement measurement. The method is currently the only way in which high resolution strain data can be obtained on flat and curved surfaces without the need for vibration isolation. [Gielisse]

A new technique to characterize superconductors with extremely low magnetic fields has been developed. [Chow]

Device Related Performance

A magnetometer based on 123 has been built and is now exhibiting 50 picotesla sensitivity for bulk material and 1 nanotesla sensitivity for screen printed thick films at 77K. This is one of the first operating devices utilizing a bulk high temperature superconductor, and exceeds the sensitivity of conventional magnetometers. As an application example, this performance would be adequate for magnetocardiograms. [Gielisse]

Weak links have been produced by striped laser irradiation across a 150 μ wide film of 123. Rf detection of 10MHz-100MHz signals has been observed confirming the Josephson like behavior of these device structures. Video detector performance is being determined. [Kleinhammes]

Passive microwave performance of our non-line-of-sight laser ablated films by Sarnoff Labs matches the best seen and gives cavity Q's of 10^3 - 10^4 at 77K for frequencies between 2-12 Ghz. This is one to two order of magnitude better than that of Cu. [Chang]

The photoresponse of high- T_c thin films was measured in the infrared, using synchrotron radiation from the Light Source at Brookhaven. A fast (~nanoseconds) bolometric component to the photoresponse, governed by the flow of heat from the film to the substrate, was discovered. The response was largest at temperatures near T_c , which implied a bolometric response. These detectors showed fast response at wavelengths from the far infrared through the visible. [Tanner, Carr]

The secondary electron emission yields from ceramic superconducting polycrystalline samples of $\text{YBa}_2\text{Cu}_3\text{O}_7$ and $\text{Pr}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-y}$ have been measured for the first time. Both materials possess very promising emission yields between 1.4-1.6 in the important low-voltage region of 0.6-0.8kV. These values match those of platinum which is widely used for cathode targets in microwave tubes. [Gielisse]

A nondestructive method to switch bulk 123 from a high J_c to a low J_c state by current pulsing has been established. The low J_c state is retained after the completion of the current pulse but can be

"erased" by reversing the direction of the current pulse (or taking the sample above T_c). Applications potential for these switching, storage, and erasure effects are being reviewed. A patent has been applied for. [Hascicek]

Measurements of magnetization and demagnetization for an oscillating ($180^\circ/1$ sec) bulk, high T_c superconductor suggest the presence of a rotational dependence for flux flow. Further work, is planned to establish if angular accelerometer device potential exists. [Rey]

Noise measurements of bulk, c- and a- axis films show enhanced spectral density and width for a-axis films, and $1/f^2$ behavior in the superconducting transition when the temperature was changing. This may impact bolometric applications. [Chen]

Field induced dissipation leading to 10% losses in magnetization cycles of 123 up to 3kOe at 4.2K has been found in 123. This may limit performance in some applications. [Rey]

A symmetry forbidden laser induced voltage has been found in 123 films. The algebraic sign of the voltage is found to be determined by the direction of the incoming light. [Kleinhammes, Foster, Hilinski]

Polymer coatings on 2122 single crystals that reduce losses by 85% have been reported. These coatings can function to enhance bolometric performance. [Schlenoff]

Developed and characterized dielectrics from the Y-Ba-Cu-O phase diagram compatible with cofiring of (123) superconductors into multilayer capacitors; (211) was chosen due to its stability. Methods were developed using porosity to control the dielectric constant. The work was extended into thick film materials compatible with thick film processing to fabricate complex three dimensional structures. [Jones]

The patterning of the thin films YBCO and BSCCO compounds was performed using the standard photolithographic technique in conjugation with a wet etching process. Successful patternings were achieved using photosensitive polyimide materials on high T_c films. A new electrochemical etching technique has been designed to etch high T_c materials. Controlled etching has been achieved by this technique using acid dilutions as high as 1:200 with water on YBCO materials. [S. Sundaram]

Theory

The coupled-cluster method has been applied to the single-hole state Hubbard model. The energy dispersion relations appear to be in good agreement with the exact diagonalization results. [Lo]

The spin-1/2 antiferromagnetic Heisenberg model on a square lattice has been used to describe the dynamics of the spin degrees of freedom of undoped copper oxides. Good agreement with experimental determinations of the spin correlation length and other properties have been obtained. The variational Monte Carlo method has been used to calculate the ground state and spin excitations of the spin-1/2 antiferromagnetic on a square lattice, and is being used to calculate the single-hole excitation spectrum in a quantum antiferromagnet. [Manousakis]

Exact solutions of the two-hole and hole-magnon bound states have been found in a quantum ferromagnet with hole hopping. Using the newly developed linked-cluster method and multiplesite Wick reduction theorem, the perturbation series of free energy, uniform susceptibility and stagger

susceptibility in the Hubbard model have been calculated up to sixth order which can help solve the Hubbard Model more accurately. [Wang]

X α program has been vectorized for ETA¹⁰ and CRAY YMP with electron exchange, and overlapping spheres, mass-velocity and spin-orbit included. The local electronic structure of the rare earth atom and the surrounding copper-oxygen network in the high-temperature superconductors RBa₂Cu₃O₇ with R=Pr, Nd, Gd, Er, and Lu has been investigated with relativistic X α scattered-wave calculations. Densities of states have been calculated for YCu₈O₈, LaCu₈O₈, and PrCu₈O₈. [Weatherford, Jones]

A first principles calculation of the electronic correlation (Hubbard U) in high T_c superconductors is being carried out using the QKKR method. [Faulkner]

A new functional integral approach to the single band Hubbard model with strong on-site repulsion U has been developed. The approximate single particle Green's function can be calculated for arbitrary U. [Levine, Muttalib]

A microscopic treatment of Gutzwiller correlations is being developed. The theory of Kotliar and Ruekenstein has been modified to make it manifestly spin rotation invariant. [Levine, Muttalib]

A slave boson representation of the projected Hubbard model is being calculated. [Wolfe]

Analysis of tunneling between an unconventional and a conventional superconductor has been carried out. There are three possible channels for tunnelling: (1) direct coupling, (2) proximity effect, and (3) when the unconventional superconductor is d-wave, it carries a temperature dependent s-wave component and tunnelling to that is possible. As a consequence of competition between these mechanisms, the temperature dependence of the critical current is found to be non-monotonic.

We have studied the electromagnetic response of an anisotropic superconductor and in the process also calculated all of the collective modes.

In an extensive study of vortices in a superconductor, we have studied the transition in a superlattice structure consisting of a superconductor-insulator sandwiches. The properties near the transition can be understood in terms of the thermally nucleated vortices. In a separate problem, we have also studied the pinning dependent localization length of vortices in a bulk superconductor. [Kumar]

The thermodynamic properties of the Y-Ba-Cu-O family of superconductors, including the parent YBa₂Cu₃O₇ compound, those derived from it by substitution of other metals for Cu, and the intercalated Y₂Ba₄Cu₇O₁₅ and YBa₂Cu₄O₈ materials has been determined. Important technological properties of those materials were predicted and were later verified experimentally, e.g. the enhanced flux pinning potential of the doped and intercalated structures. Other predictions include the existence of materials that have only recently been observed in thin films, notably the YBa₂Cu₅O₉ structure-a compound that may have totally new superconducting properties. [Wille]

Distributed Center Program

Finally, we list as an accomplishment set at high priority, the achievement of a distributed center program in a state university system of seven campuses, involving over 50 principal investigators. Participating in the cooperative sharing were over 30 faculty members who individually visited different institutions (some for stays of up to 1 year) to transfer knowledge, to attend statewide meetings of program scientists, and to share fundamental understanding, measurement techniques, and samples. This rapid broadening of the knowledge base through personal familiarity could not have occurred prior to the DARPA/SUS-sponsored Advanced Microelectronics and Materials Program.

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Advanced Composite Materials

The overall objective of this program was to provide a fundamental understanding of the processing science and technology necessary to fabricate ceramic-matrix, intermetallic-matrix, and metal-matrix composites with superior mechanical properties in high temperature and oxidizing environments. The composites are intended for use as structural materials for advanced aerospace applications at temperatures exceeding 1200°C (2200°F).

In order to accomplish the program objective, interactive research groups were established in four key areas:

- | | |
|-----|--------------------------------|
| I | Fiber Fabrication |
| II | Coatings and Infiltration |
| III | Composite Fabrication |
| IV | Physical/Mechanical Properties |

The objective of the fiber fabrication group was to develop new fibers which have superior strength and toughness at temperatures above 1200°C in oxidizing environments. The research effort focused on the development of three types of fibers: (1) glass-free mullite-based fibers, (2) stabilized zirconia-based fibers, and (3) oxygen-free silicon carbide fibers. The coatings program had two primary objectives: (1) to control the characteristics of matrix/reinforcing phase interfaces (e.g., to control chemical reactions and bonding at a matrix/fiber interface) and (2) to develop coatings that will improve the oxidation resistance of metal-matrix and intermetallic-matrix composites. Coatings methods utilized included chemical vapor deposition, sol-gel processing, and solution coating with polymeric precursors to ceramics.

The composite fabrication group investigated various methods to incorporate reinforcing phases (i.e., fibers, whiskers, and particulates) into ceramic-, metal-, and intermetallic-matrices. The objectives were not only to utilize innovative processing techniques, but also to develop an improved scientific understanding of processing-microstructure relationships in composites fabrication. Processing methods investigated included colloidal processing, chemical vapor infiltration, reactive hot-compaction and *in situ* coating, and microwave sintering.

The objectives of the physical properties program were to evaluate mechanical properties of the composites produced, to investigate phase stability at high temperatures, and to establish microstructure-mechanical property relationships for the composite materials. As part of a fundamental approach to the microscale design of high temperature composite materials with tailored physical properties, this program also used mathematical modeling to study the behavior of the composites.

Some of the highlights of the accomplishments are listed below, with additional details given later and in the publications and patents listed at the end of this report.

- High-molecular weight polycarbosilanes have been synthesized which, upon pyrolysis, give a high-yield of silicon carbide with low oxygen contamination. The fibers show significant properties improvement compared to those available commercially.

- A novel process known as Transient Viscous Sintering (TVS) has been developed and applied toward the development of mullite-based composites.
- A novel reactive hot compaction and *in situ* coating technique has been developed to produce niobium-based intermetallic matrix composites.
- Short fibers of glass-free mullite with high relative density and fine grain size have been fabricated using sol-gel processing. Composite fibers containing ~10 vol% tetragonal zirconia have also been produced.
- Microwave energy has been demonstrated as a viable method for ignition of self-propagating synthesis.
- A process has been developed for pressureless sintering of alumina/silicon carbide whisker and alumina/zirconia/silicon carbide whisker composites with whisker concentrations as high as ~27 vol%.
- Chemical vapor deposition (CVD) of titanium carbide onto substrates and chemical vapor infiltration (CVI) of the carbide into fiber preforms have been carried out. The feasibility of using atomic layer deposition (ALD) technique for producing thin coatings has been demonstrated.
- Polysilazanes have been synthesized which, upon pyrolysis, give a high-yield of silicon carbide/silicon nitride. Adherent coatings have been produced on several substrate materials.
- Ytria-stabilized tetragonal zirconia with dispersed alpha-alumina has been produced in short-fiber form using sol-gel processing. Microstructural characterization shows that fibers have high relative density (i.e., low porosity) and submicrometer grain size.
- Ductile phase reinforced composites with tantalum-based alloy and molybdenum disilicide matrices with improved fracture toughness have been produced by hot compaction.
- The effect of microstructure on mechanical properties of selected Nb-Ti-Al intermetallics has been evaluated.
- A finite element technique for evaluating the effect of cracks in the vicinity of bimaterial interfaces on the overall fracture toughness has been developed.

Fiber Fabrication

Sol-Gel Processing of Mullite-Based Fibers

The main objective of this investigation was to prepare mullite fibers which contain no glass-forming additives (e.g., boria). Another objective was to demonstrate the feasibility of producing composite mullite/zirconia fibers. We have successfully fabricated mullite and mullite-zirconia fibers using three types of sols: aluminum formoacetate/colloidal silica (method #1), aluminum chlorohydroxide/colloidal silica (method #2), and aluminum butoxide/silicon ethoxide (method #3).

A laboratory scale apparatus for continuous spinning of sol-gel produced fibers has been constructed. Mullite fibers produced by method #3 have shown the best spinnability of the various sols tested to this point. Although long fiber lengths could be obtained using the continuous spinning apparatus, fibers were too brittle to be wound continuously.

Fibers with high relative density (low porosity), fine grain size, and complete conversion to mullite could be produced after heat treatment in the range 1200-1500°C. Even after heat treatment at 1400°C, the average grain size is considerably less than 1 μm . Detailed characterization of the microstructure evolution has been carried out for fibers produced using differential thermal analysis, thermal gravimetric analysis, nitrogen gas adsorption (for specific surface area and pore size distribution), X-ray diffraction, and scanning and transmission electron microscopy. In addition, the fibers have been tested for chemical compatibility with intermetallic matrices. [Venkatachari, Sacks et al., 1,2]

Processing of Zirconia-Based Composite Fibers

Yttria-stabilized tetragonal ZrO_2 polycrystals (Y-TZP) exhibit very high strength and toughness. It has been reported that the addition of Al_2O_3 increases both room- and high-temperature strengths of Y-TZP. Despite these excellent attractive properties, ZrO_2 composite fibers have never been prepared. The main objective of this research was to develop ZrO_2 -based composite (Y-TZP/ Al_2O_3) fibers whose mechanical properties are superior to the existing oxide fibers at temperatures above 1200°C in an oxidizing atmosphere.

Spinnable sols containing colloidal ZrO_2 , yttrium and aluminum precursors and polymer were prepared and discontinuous fibers with diameters ranging from 5-15 μm were fabricated. The processing conditions for fiber spinning have been established. Variables affecting rheological properties of the sols were investigated by viscometry and Fourier transformation infrared spectroscopy.

The Y-TZP/ Al_2O_3 fibers having composite microstructures with >99% relative density were produced by sintering up to 1500°C. These microstructures consist of elongated Al_2O_3 grains dispersed in a fine-grained tetragonal ZrO_2 matrix. The elongated Al_2O_3 particles were 0.3 μm wide with an aspect ratio >3 and randomly distributed with a localized grain alignment at 1500°C.

Thermal stability of the fibers was studied at temperatures 1400° and 1500°C for prolonged periods of time. The Al_2O_3 addition enhanced the high-temperature microstructural stability of the fibers with respect to exaggerated grain growth of ZrO_2 . Large cubic ZrO_2 grains were found on the Y-TZP fiber surfaces tested above 1400°C, but not on the Y-TZP/ Al_2O_3 fiber surfaces tested under identical conditions. Addition of 15 wt% Al_2O_3 inhibited grain growth of ZrO_2 on the fiber surfaces at temperatures up to 1400°C. Above this, Al_2O_3 is less effective due to its own coarsening. [Sim, Clark et al., 3-5]

Coatings and Infiltration

Polymeric Precursors to Ceramic Materials

The research program on preceramic polymers has focused on the identification and development of new and useful preceramic polymer systems for producing fibers and/or coatings. Efforts were

concentrated on two areas: the development of a novel form of polycarbosilane for the preparation of silicon carbide fibers, and development of vinylic polysilazanes for the preparation of silicon carbide and/or silicon nitride coatings. For the former project, high-molecular weight polycarbosilanes were synthesized which, upon pyrolysis, gave a high yield of silicon carbide with low oxygen contamination. There is much evidence that the presence of SiO_2 in the polycarbosilane-derived fibers such as Nicalon® leads to detrimental effects on the high temperature mechanical properties. In view of this, the main objective of this project was to produce polymer-derived SiC fibers containing little or no SiO_2 .

We have developed a new high MW polycarbosilane, which can be successfully dry-spun (solution spun) into fibers. These fibers, since they do not melt when heated, could be directly pyrolyzed to silicon carbide, thus eliminating the oxidative crosslinking step. The resulting SiC fibers were found to contain no SiO_2 (less than 1%) and have superior high temperature mechanical properties. The new polycarbosilane gave a ceramic yield of approximately 80% which is equal to that of oxidized conventional polycarbosilane. In addition to having potential as a precursor to oxygen-free SiC fibers, the new high MW polycarbosilane has been used to prepare uniform coatings on alumina substrates.

A small-scale fiber-spinning apparatus was assembled to determine the processing characteristics of potential fiber precursors. Using this apparatus we have been able to spin continuous fiber polycarbosilane. These fibers have been pyrolyzed to 1000°C to yield continuous fibers with diameters ranging from 10 to 20 μm . Such fibers were shown to have significantly higher retention of mechanical strength after heat treatments than do either Nicalon or HPZ fibers. For example, average tensile strengths of greater than 0.9 GPa after heat treatment at 1700°C have been demonstrated.

For the second project, the vinyl polymerization of 1,3,5-trimethyl-1,3,5-trivinylcyclotrisilazane (135T) was studied. A vinylic polysilazane was developed which gives a high yield of SiC/Si₃N₄ ceramic upon pyrolysis. This material was used to produce adherent coatings on a variety of substrates, and also infiltration/densification of porous ceramics. Pyrolysis of poly(135T) to 1000°C in a nitrogen atmosphere gives a 65% yield of black ceramic material, which was found to contain both α and β Si₃N₄ (the β Si₃N₄ was in the form of whiskers) as well as amorphous material. The material showed excellent stability towards oxygen at 1500°C . Further investigations into the basic chemistry of this system, the pyrolysis process, and the characterization of the resulting ceramics are in progress. [Toreki, Batich et al., 6-10; Patents 1-3]

Chemical Vapor Deposition and Infiltration

In this project, chemical vapor deposition was used to deposit TiC_x coating on a variety of substrates (Al_2O_3 , Ta, Nb, Nicalon, SiO_2 , graphite, ZrO_2 , sapphire and Mo). The growth of the carbide was found to be limited by heterogeneous reaction to a temperature of 1200°C . Moreover, the morphology and grain orientations were strongly influenced by substrate type and growth temperature. It was also found that whisker growth could be promoted with some of the substrates, thus providing an opportunity to improve the mechanical properties of the coating.

The microhardness and fracture toughness of TiC_x on several substrates was measured. In the temperature range 1050 - 1200°C , the hardness of TiC_x increased with increasing deposition temperature while the fracture toughness decreased. This behavior was attributed to the formation of highly stressed films grown at high growth rates and to the presence of interstitial carbon.

These results suggest that an optimal deposition temperature exists for growth of films with improved mechanical properties.

In addition, atomic layer deposition (ALD) technique was used to produce thin coatings of controlled composition. This technique takes advantage of the difference in adsorption energies between physical and chemical adsorption for deposition of TiC_x . In this process, growth with atomic level control is achieved by sequentially exposing a substrate to TiCl_4 , then H_2 , then CH_4 , then H_2 , and repeating the process. One of the most important results of this program was the first demonstration of this process in a ceramic system (TiC_x on Al_2O_3). A dramatic reduction in growth temperature was also demonstrated (TiC_x does not deposit below $\approx 1000^\circ\text{C}$ in conventional CVD but this process was performed at 500°C). Because of the low growth temperature, processes previously limited by mass transfer become kinetically limited with the promise of extremely uniform coatings.

Chemical vapor infiltration studies were also performed on stacks of fiber weaves. A novel radiantly cooled fiber weave holder was designed to establish a large temperature gradient, and the infiltration of process was investigated as a function of temperature gradient, reaction flow rate and gas phase composition. Remarkable levels of uniform infiltration were possible if the maximum deposition temperature was maintained below 1100°C . The TiC_x infiltrated Si-C-O weaves were characterized for grain orientation, morphology, microhardness and fracture toughness. [Anderson, Sacks et al., 11-14]

Composite Fabrication

Pressureless Densification of Ceramic-Matrix Composites

Colloidal and Infiltration Processing of Whisker-Reinforced Composites

Recent studies have shown that vastly improved densification can be achieved by using green compacts with homogeneous microstructure, fine pore sizes, and high relative density. We have produced alumina/SiC whisker and alumina/zirconia/SiC whisker compacts with these characteristics using colloidal and infiltration processing techniques. Variables that have been investigated include the effects of (i) matrix particle size, (ii) liquid phase sintering additives, (iii) aspect ratio of the SiC whiskers, and (iv) infiltration additives. Some of the key results are briefly summarized below.

Suspension processing was used to prepare homogeneous Al_2O_3 /SiC whisker compacts with high green density (~ 69 - 71%) and small median pore channel radius (≤ 25 nm). Pressureless sintering behavior was investigated for compacts prepared with 0-30 vol% SiC whiskers. It was possible to enhance densification by using Al_2O_3 powders with higher specific surface area (27 m^2/g vs. 10 m^2/g). For example, samples with 5 vol% whiskers could be sintered at only 1250°C (30 min) to $\sim 97\%$ relative density (with zero open porosity and ~ 0.28 μm average grain intercept size). With 15 vol% whiskers, it was necessary to sinter at 1800°C (30 min) to reach the same relative density. The average grain intercept size increased to ~ 1.6 μm .

Densification could be enhanced in samples with high whisker concentrations (i.e., >15 vol%) by increasing the compact green density via an infiltration technique. Molten aluminum nitrate was infiltrated into green compacts and subsequently decomposed to aluminum oxide by low temperature heat treatment. The green relative density was increased to $\sim 80\%$ using several infiltration cycles. Samples with ~ 27 vol% SiC whiskers could be pressureless sintered to $\sim 94\%$

relative density. Fracture toughness measurements on infiltration-processed samples showed similar increases in fracture toughness (with increasing whisker content) as observed for samples without infiltration. [Sacks and Lee, 15-17; Patents 4-6]

Transient Viscous Sintering

Recent studies of composite sintering behavior have shown that reinforcing phases exert less constraint on densification when the matrix phase is amorphous (non-crystalline) and can deform viscously under the sintering stress. Despite the beneficial effect on densification, amorphous phases are usually avoided in fabricating materials for high temperature applications because mechanical properties (e.g., strength, creep resistance, etc.) are adversely affected by the presence of phases that deform viscously under an applied stress. A process known as Transient Viscous Sintering (TVS) has been developed at the University of Florida in order to take advantage of viscous sintering for enhanced densification, but to avoid the adverse effect of amorphous phases on mechanical properties. The process involves the synthesis of submicrometer particles (using a solution technique) which have a composite structure. The particles have a core of one material (alumina) and an outer layer of another material (amorphous silica). The outer layer allows the particles to densify by viscous flow during sintering. However, the amorphous silica is a transient phase. At temperatures above the sintering temperature, the core and outer layer react to form the crystalline compound mullite. This process was used to produce high density, fine-grained mullite samples.

The TVS process can readily be adapted to form mullite-based composites by utilizing other core particles (i.e., in addition to alumina). For example, composite powders with cores of zirconia, silicon carbide, and silicon nitride have been synthesized. These composite powders are then used to prepare high density, fine-grained mullite and mullite-based composites (e.g., mullite/zirconia, mullite/SiC whiskers, etc.), silicon nitride-based composites, and SiAlON-based composites. Mechanical property measurements have been initiated on samples prepared by the TVS process. The initial results show that mullite samples retain their room temperature flexural strength to at least 1400°C. [Sacks, Scheiffele, Bozkurt et al., 18-22; Patents 7-8]

Metal and Intermetallic Matrix Composites

The objective of this research program is to synthesize metal- and intermetallic-matrix composites for structural use at temperatures above 1200°C. Three classes of alloys were selected for investigation: (1) Nb-base aluminides with the major focus on NbAl₃, (2) Ta-base ternary aluminides with the major focus on TaTiAl₂, and (3) High temperature silicides with the major focus on MoSi₂. Although other intermetallics were considered, the focus on these materials was motivated by the expectation that the inherent advantages of these matrices can be exploited effectively via artificial compositing, targeted specifically to overcome their disadvantages. For example, NbAl₃ has high specific modulus and strength (density 4.62 g/cc), oxidation resistance which is the best among all Nb-aluminides, but poor ambient temperature fracture toughness (~2 MPa·√m). TaTiAl₂ on the other hand has good high temperature oxidation resistance and better toughness (~8 MPa·√m), but inadequate specific strength and susceptibility to high temperature creep. Among the advantages of MoSi₂ are a high melting point (above 2000°C) and excellent oxidation resistance, the disadvantage being the poor ambient fracture toughness. The compositing strategy was targeted towards improving the fracture toughness of NbAl₃ and MoSi₂ via ductile phase toughening and the strength and creep resistance of TaTiAl₂ via hybrid compositing with high strength reinforcements.

NbAl₃ Matrix Composites

Reactive hot compaction (RHC) has been successfully utilized to produce NbAl₃ matrix with close to 100% theoretical density at relatively low processing temperatures of around 1300°C. In addition to NbAl₃, NbAl₃ matrix composites containing a dispersion of Nb₂Al or Nb₃Al particles with a niobium core were also produced via RHC by controlling the initial stoichiometry. The fracture toughness of the particle dispersed matrix was determined to be 3.5 MPa·√m, compared with 1.9 MPa·√m for the monolithic matrix prepared by conventional techniques. The improvement is due to the fact that in the particle-dispersed matrix, cracks tended to become arrested and/or deflected at the second phase particles.

In order to further improve the ambient temperature fracture toughness of NbAl₃, composites of the intermetallic reinforced with ductile Nb filaments were produced. To produce the alumina coatings, a unique coupling of RHC and reinforcement pretreatment was utilized. Specifically, Nb filaments were pre-oxidized at 500°C and cold compacted with elemental powders of Nb and Al. During the subsequent hot pressing, the surface oxides on these Nb filaments react with aluminum to form a continuous alumina coating. At the same time, the elemental powders convert to the intermetallic matrix. The coating thickness was varied from 3 to 8 μm by varying the oxidation time of the Nb filaments.

Long term annealing at 1200°C indicated the *in situ* Al₂O₃ coating to be stable at these temperatures and to serve as an effective reaction barrier. Moreover, the coating imparted appropriate bonding characteristics to improve fracture toughness of the composites. For example, composites containing 20 vol.% Nb filaments had fracture toughness five times higher than the matrix. Fracture surface analysis indicated that partial decohesion had occurred at the filament/matrix interfaces allowing the Nb filaments to fail in a ductile manner. Therefore, the *in situ* alumina coating not only performs as an effective diffusion barrier, but leads also to desirable interface strengths from a ductile fiber toughening standpoint. Various other aspects of RHC processing of other intermetallics, the coupling between RHC and reinforcement pretreatment to produce interface diffusion barrier coatings "*in situ*", and microstructural analysis and mechanical testing of the composites were studied, as detailed in the publications listed at the end of the report. [Abbaschian, Kaufman et al., 23-27; Patent 9]

MoSi₂ Matrix Composites

The chemical compatibility between MoSi₂ and various ductile intermetallics, refractory metals and oxide ceramics were examined. Among oxide ceramics, alumina and the mullite fibers produced in this program were found to be compatible with MoSi₂ provided no silica was present in the matrix.

In the presence of silica, a spinel type reaction product was produced at the boundary between alumina or mullite and MoSi₂.

Based on these results, a series of MoSi₂ composites, containing various volume fractions of alumina coated Nb filaments was produced. Fracture toughness tests were performed on chevron-notched samples using four-point bending. For example, the fracture toughness of MoSi₂ containing 30 vol% uncoated Nb filaments showed a four-fold increase in toughness as compared with that of the unreinforced matrix, while coated Nb filaments improved the fracture toughness of the composites even further. Fracture surface analysis of the MoSi₂ matrix composites showed that the matrix failure was primarily intergranular in nature with a significant amount of a second phase (or phases) at the grain boundaries.

The second phase particles in the compacts were found to be primarily amorphous silica, Mo_5Si_3 and CMo_5Si_3 ; these were found to originate from a combination of both the silica that forms on the surfaces of the powders, which becomes incorporated into the matrix upon consolidation, and the reaction of interstitial impurities with silicon to form silica plus other intermetallic phases. Thus, alloying additions and processing parameters were varied in an attempt to reduce and/or control the types and distributions of second phases in the MoSi_2 . For example, when carbon was added to MoSi_2 and compacted at sufficiently high temperatures (above 1500°C), it reduced the silica to form various reaction products - Si, SiO , CO, CO_2 , and SiC - depending on the actual processing variables. Since three of the five products listed are gaseous, it was found that one must use processing conditions in which the gas can escape from the sample, i.e., vacuum hot pressing as opposed to hot isostatic pressing. Otherwise, internal gas bubbles form and result in substantial swelling of the sample.

Alternative approaches for reducing the SiO_2 on the powder surfaces prior to or during consolidation are also being investigated. For example, aluminum has been added to MoSi_2 prior to mechanical alloying in an effort to transform the silica to something more desirable such as alumina or mullite. SEM examination of these samples show a complete absence of the Mo_5Si_3 (or CMo_5Si_3) phases. X-ray diffraction peaks are consistent with mullite, and TEM analysis is being attempted to confirm the identification.

In an effort to further elucidate the effect of these second phase particles on the properties of the composites, mechanical alloying (MA) was used to produce MoSi_2 with and without submicron ceramic dispersoids. The compressive strengths of compacts made with the mechanically alloyed materials were superior to those made from commercial powders and the influence of the ceramic dispersoids were determined. In addition, the microstructures of the MA compacts appeared to contain considerably less second phase particles than the commercial compacts when processed at low temperatures. [Abbaschian, Kim, Xiao, Kaufman et al., 28-38]

Ta-Ti-Al Composites

The phase equilibria and transformations of Ta-Ti-Al intermetallic alloys have been studied in some detail. A 1450°C isothermal section containing seven equilibrium two-phase fields ($\alpha+\beta$, $\alpha+\gamma$, $\gamma+\sigma$, $\alpha+\eta$, $\eta+\text{Al}_2\text{Ta}$, $\beta+\sigma$, $\eta+\text{AlTa}$) and considerable extension of the AlTa and Al_2Ta phases into the ternary section has been constructed. In addition, discrepancies concerning the crystal structure of the binary Al_2Ta phase (complex fcc) have been resolved and the nature of the stacking faults in this phase have been identified. Finally, the presence of an intermediate stable ordered B2 phase has been observed to exist at 1450°C in disagreement with previous reports by other authors.

The phase transformations in the intermediate alloys containing ~50% Al, which are disordered β at 1450°C, are complex and strong functions of cooling rate and initial structure. For example, it has been shown that the high temperature disordered β phase can decompose via a number of reactions as described in the publications listed at the end of this report. Consequently, it is possible to produce a relatively wide range of transformation structures although it is necessary to exercise caution when interpreting the manner in which these microstructures evolve and their corresponding thermal stabilities.

Concurrently, hybrid composites of TaTiAl_2 reinforced with FP fibers and Nb filaments were produced by hot compaction in vacuum. The composites showed improved ambient temperature toughness because of the ductile Nb reinforcements and improved high temperature strength because of the ceramic fibers. [Kaufman, Weaver et al., 39-41]

Microwave Processing of Composites

This project involves three major thrust areas: (1) microwave sintering, (2) microwave combustion, and (3) microwave enhanced solid state reactions. To apply the benefits of ultra-rapid heating to MW sintering, a technique was developed that makes use of both MW-material interaction and conventional radiant/conduction heating mechanisms. This technique has been designated as "microwave hybrid heating" and makes use of radiant/conduction heating to rapidly heat samples through low temperature regimes to the critical MW interaction temperature (800°C for pure alumina). This facilitates very rapid heating at the low frequency of 2.45 GHz. For example, heating rate of up to 1500°C in 120 sec. was achieved for pure alumina in a commercially available microwave oven.

This technique also resulted in an accelerated densification process, compared to conventional fast firing, and to culminate in better microstructural homogeneity relative to conventional fast firing or stand-alone MW sintering. It is expected that the improved microstructural homogeneity translates into enhanced and more uniform mechanical properties.

Microwave energy also was used to ignite self-propagating high-temperature synthesis (SHS) reactions. Numerous ceramics and composites have been prepared successfully. These include $\text{Al}_2\text{O}_3/\text{TiC}$, $\text{Al}_2\text{O}_3/\text{TiB}_2$, TiC/TiB_2 and $\text{Al}_2\text{O}_3/\text{SiC}$. However, since the density of the product is higher than that of the reactants, porosity is observed in the bulk sample. More work is required to reduce this porosity.

Solid state reactions between Al_2O_3 and ZnO have been carried out in the microwave furnace. The results indicate enhanced diffusion and reaction between these materials in the microwave furnaces as compared to the conventional furnace. [Clark, Dalton, Ahmad, Dé et al., 42-50; Patents 10-11]

Physical/Mechanical Properties

Crack/Interface Interaction and Composite Fracture Toughness

The objectives of the present study are to: (i) develop a failure criterion for near-interface cracks based on the non-singular but very high stress concentrations along the interface; (ii) express the criterion in terms of an unconventional stress intensity factor for the problem of a crack touching the interface; (iii) explore the adequacy of the calculations by testing some model bimaterial specimens; and (iv) numerically simulate crack propagation in a model composite to estimate the macroscopic toughness of the composite from the matrix, reinforcement and interfacial properties.

The research on the influence of interfacial properties on composite mechanical properties focused on the development of the analytical and numerical tools necessary to model the failure processes of interest: cracks in the vicinity of bimaterial interfaces. Finite element methods of conventional type and a displacement discontinuity method have been used to analyze several interface/crack problems, including a bimaterial beam specimen to be used for model experiments. In addition, a beam finite element has been developed to perform thermo-elastic analysis of bimaterial beams and delaminated composites.

A method which has the potential to produce precise results with moderate computational effort is the displacement discontinuity method. This method, which involved a boundary element formulation based on a dislocation-related elasticity solution, has been implemented for bimaterial problems of interest and employed to solve several crack problems. The details of the numerical approach are published elsewhere, along with some of the results for a crack in an infinite bimaterial medium. The precise results concerning stress distributions, singularities and energy release rates which are obtainable from the displacement discontinuity method are essential to the development of a comprehensive model of crack/interface failure processes. [Sankar et al., 51-53]

Development of Intermetallic Matrices

The objective of this project is to design and to develop multiphase intermetallic materials that have reasonable room temperature toughness and high temperature creep resistance. The approach taken included (i) an evaluation of the phase stability (or instability) through heat treatment and microstructural characterization, (ii) a study of the fracture toughness of developed microstructures and identification of toughening mechanisms, (iii) an investigation of the creep resistance of the developed microstructures at elevated temperatures, and finally (iv) an integration of results of these studies for modification of microstructures through the control of chemical composition and heat treatment. We have chosen Nb-Ti-Al ternary system for this investigation.

Our studies on stability of alloys with compositions toward the central portion of this system have shown that microstructures consisting of $\sigma+\beta$, $\sigma+\gamma$, and $\sigma+\gamma+\beta$ can be produced which are very stable (i.e., no grain growth, precipitation, Ostwald ripening, etc.) at temperatures as high as 1300°C. Although the $\sigma+\gamma$ microstructure is stable upon slow cooling, β may transform to other phases, such as the orthorhombic phase and the ω -type phase. The toughness of the above mentioned microstructures have been evaluated using various methods including microhardness indentation and chevron-notched bend specimens. The high temperature strength of the developed alloy was measured using compression testing. The results of the mechanical and microstructural studies suggest that the developed alloys have promising properties for use at high temperature structural applications. [Ebrahimi, Hoelzer, Morrone et al., 54-59]

Advanced Microelectronics and Materials Program

Summary Reports and Presentations

1. **Advanced Microelectronics and Materials Program Annual Report for the period July 1, 1988 to September 30, 1989.**
2. **Advanced Microelectronics and Materials Program Annual Review Presentation, January 16-18, 1990.**
3. **Advanced Microelectronics and Materials Program Annual Review Presentation, October 16-18, 1990.**
4. **Advanced Microelectronics and Materials Program Annual Report for the period October 1, 1989 to December 31, 1990.**

AMMP Newsletters

1. **AMMP Newsletter, Vol. 1, No. 1, January 1990.**
2. **AMMP Newsletter, Vol. 1, No. 2, March 1990.**
3. **AMMP Newsletter, Vol. 1, No. 3, May 1990.**
4. **AMMP Newsletter, Vol. 1, No. 4, July 1990.**
5. **AMMP Newsletter, Vol. 1, No. 5, September 1990.**
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